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NEWS	2	JAN 02	STN pricing information for 2008 now available
NEWS	3	JAN 16	CAS patent coverage enhanced to include exemplified prophetic substances
NEWS	4	JAN 28	USPATFULL, USPAT2, and USPATOLD enhanced with new custom IPC display formats
NEWS	5	JAN 28	MARPAT searching enhanced
NEWS	6	JAN 28	USGENE now provides USPTO sequence data within 3 days of publication
NEWS	7	JAN 28	TOXCENTER enhanced with reloaded MEDLINE segment
NEWS	8	JAN 28	MEDLINE and LMEALINE reloaded with enhancements
NEWS	9	FEB 08	STN Express, Version 8.3, now available
NEWS	10	FEB 20	PCI now available as a replacement to DPCI
NEWS	11	FEB 25	IFIREF reloaded with enhancements
NEWS	12	FEB 25	IMSPRODUCT reloaded with enhancements
NEWS	13	FEB 29	WPINDEX/WPIDS/WPIX enhanced with ECLA and current U.S. National Patent Classification
NEWS	14	MAR 31	IFICDB, IFIPAT, and IFIUDB enhanced with new custom IPC display formats
NEWS	15	MAR 31	CAS REGISTRY enhanced with additional experimental spectra
NEWS	16	MAR 31	CA/CAPLUS and CASREACT patent number format for U.S. applications updated
NEWS	17	MAR 31	LPCI now available as a replacement to LDPCI
NEWS	18	MAR 31	EMBASE, EMBAL, and LEMBASE reloaded with enhancements
NEWS	19	APR 04	STN AnaVist, Version 1, to be discontinued
NEWS	20	APR 15	WPIDS, WPINDEX, and WPIX enhanced with new predefined hit display formats
NEWS	21	APR 28	EMBASE Controlled Term thesaurus enhanced
NEWS	22	APR 28	IMSRESEARCH reloaded with enhancements
NEWS EXPRESS	FEBRUARY 08 CURRENT WINDOWS VERSION IS V8.3, AND CURRENT DISCOVER FILE IS DATED 20 FEBRUARY 2008		
NEWS HOURS	STN Operating Hours Plus Help Desk Availability		
NEWS LOGIN	Welcome Banner and News Items		
NEWS IPC8	For general information regarding STN implementation of IPC 8		

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\* \* \* \* \* STN Columbus \* \* \* \* \*

FILE 'HOME' ENTERED AT 07:06:31 ON 13 MAY 2008

=> FILE CASREACT

COST IN U.S. DOLLARS

SINCE FILE

TOTAL

ENTRY

SESSION

FULL ESTIMATED COST

0.21

0.21

FILE 'CASREACT' ENTERED AT 07:06:55 ON 13 MAY 2008

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FILE CONTENT:1840 - 10 May 2008 VOL 148 ISS 20

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\*\*\*\*\*  
\*  
\* CASREACT now has more than 13.8 million reactions \*  
\*  
\*\*\*\*\*

Some CASREACT records are derived from the ZIC/VINITI database (1974-1999) provided by InfoChem, INPI data prior to 1986, and Biotransformations database compiled under the direction of Professor Dr. Klaus Kieslich.

This file contains CAS Registry Numbers for easy and accurate substance identification.

=>

Uploading C:\Program Files\Stnexp\Queries\ST.str product

L1 STRUCTURE UPLOADED

=>

Uploading C:\Program Files\Stnexp\Queries\ST.str reactant/reagent

L2 STRUCTURE UPLOADED

=> S L1 FULL

FULL SEARCH INITIATED 07:07:39 FILE 'CASREACT'

SCREENING COMPLETE - 23160 REACTIONS TO VERIFY FROM 5791 DOCUMENTS

100.0% DONE 23160 VERIFIED 4735 HIT RXNS

1392 DOCS

SEARCH TIME: 00.00.07

L3 1392 SEA SSS FUL L1 ( 4735 REACTIONS)

=> S L3 AND IR

33853 IR

L4 50 L3 AND IR

=> S L4 AND LITHIUM METAL

25160 LITHIUM

51803 METAL

106 LITHIUM METAL

(LITHIUM(W)METAL)

L5

1 L4 AND LITHIUM METAL

=&gt; D L5 IBIB ABS CRD 1

L5 ANSWER 1 OF 1 CASREACT COPYRIGHT 2008 ACS on STN

ACCESSION NUMBER: 143:248504 CASREACT

TITLE: Method for producing alkyl lithium compounds and aryl lithium compounds by monitoring the reaction by means of ir-spectroscopy

INVENTOR(S): Weiss, Wilfried; Dawidowski, Dirk; Pleyer, Walter; Krueckel, Frank

PATENT ASSIGNEE(S): Chemetall G.m.b.H., Germany

SOURCE: PCT Int. Appl., 32 pp.

CODEN: PIXXD2

DOCUMENT TYPE: Patent

LANGUAGE: German

FAMILY ACC. NUM. COUNT: 1

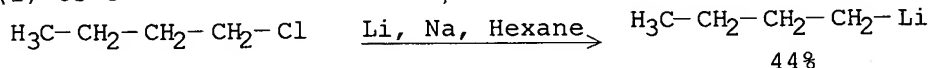
PATENT INFORMATION:

PATENT NO.	KIND	DATE	APPLICATION NO.	DATE
WO 2005082911	A1	20050909	WO 2005-EP1954	20050224
W:	AE, AG, AL, AM, AT, AU, AZ, BA, BB, BG, BR, BW, BY, BZ, CA, CH, CN, CO, CR, CU, CZ, DE, DK, DM, DZ, EC, EE, EG, ES, FI, GB, GD, GE, GH, GM, HR, HU, ID, IL, IN, IS, JP, KE, KG, KP, KR, KZ, LC, LK, LR, LS, LT, LU, LV, MA, MD, MG, MK, MN, MW, MX, MZ, NA, NI, NO, NZ, OM, PG, PH, PL, PT, RO, RU, SC, SD, SE, SG, SK, SL, SM, SY, TJ, TM, TN, TR, TT, TZ, UA, UG, US, UZ, VC, VN, YU, ZA, ZM, ZW			
RW:	BW, GH, GM, KE, LS, MW, MZ, NA, SD, SL, SZ, TZ, UG, ZM, ZW, AM, AZ, BY, KG, KZ, MD, RU, TJ, TM, AT, BE, BG, CH, CY, CZ, DE, DK, EE, ES, FI, FR, GB, GR, HU, IE, IS, IT, LT, LU, MC, NL, PL, PT, RO, SE, SI, SK, TR, BF, BJ, CF, CG, CI, CM, GA, GN, GQ, GW, ML, MR, NE, SN, TD, TG			
DE 102004009445	A1	20050929	DE 2004-10200400944520040227	
EP 1723153	A1	20061122	EP 2005-733858	20050224
R:	AT, BE, BG, CH, CY, CZ, DE, DK, EE, ES, FI, FR, GB, GR, HU, IE, IS, IT, LI, LT, LU, MC, NL, PL, PT, RO, SE, SI, SK, TR			
CN 1922192	A	20070228	CN 2005-80005827	20050224
IN 2006CN03106	A	20070608	IN 2006-CN3106	20060825
US 20070152354	A1	20070705	US 2006-589715	20061023
PRIORITY APPLN. INFO.:			DE 2004-10200400944520040227	
			WO 2005-EP1954	20050224

OTHER SOURCE(S): MARPAT 143:248504

AB The invention relates to a method for producing alkyl lithium compds. and aryl lithium compds. by reacting lithium metal with alkyl or aryl halogenides in a solvent, the concentration of the alkyl/aryl halogenide and the alkyl/aryl lithium compound being detected according to an in-line measurement in the reactor by IR spectroscopy, and an exact recognition of the end point of the dosing of the halogenide constituents being carried out by evaluation of the IR measurement. Said method enables an optimum reactive process and reaction yield. The identification of the resp. concentration of the adduct and the product is a reliable reactive process. The yield of the reaction is also optimized by determining the end point of the halogenide dosing, as is the purity of the product due to a lower concentration thereof during the reaction.

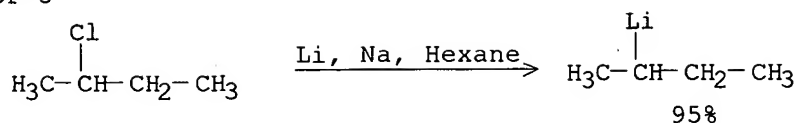
RX(1) OF 5



448

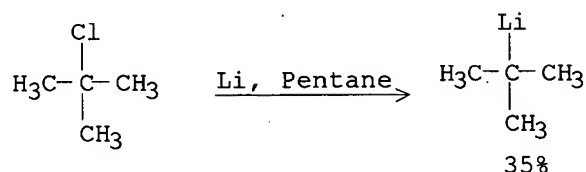
CON: 280 minutes, room temperature

RX(2) OF 5



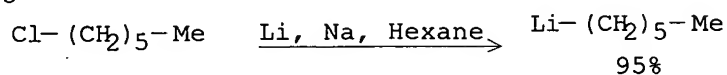
CON: 75 minutes, 40 deg C, 290 atm

RX(3) OF 5



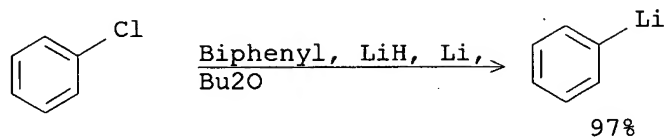
NOTE: tert-butyllithium mediated  
CON: 144 minutes, room temperature

RX(4) OF 5



CON: 40 deg C, 290 atm

RX(5) OF 5



CON: 4 hours, 35 deg C

REFERENCE COUNT: 1 THERE ARE 1 CITED REFERENCES AVAILABLE FOR THIS RECORD. ALL CITATIONS AVAILABLE IN THE RE FORMAT

=> D L4 IBIB ABS CRD 1-50

L4 ANSWER 1 OF 50 CASREACT COPYRIGHT 2008 ACS on STN

ACCESSION NUMBER: 148:192013 CASREACT

TITLE: Isomeric Forms of Heavier Main Group Hydrides:  
Experimental and Theoretical Studies of the [Sn(Ar)H]<sub>2</sub>  
(Ar = Terphenyl) System

AUTHOR(S): Rivard, Eric; Fischer, Roland C.; Wolf, Robert; Peng,  
Yang; Merrill, W. Alexander; Schley, Nathan D.; Zhu,  
Zhongliang; Pu, Lihung; Fettinger, James C.; Teat,  
Simon J.; Nowik, Isreal; Herber, Rolfe H.; Takagi,  
Nozomi; Nagase, Shigeru; Power, Philip P.

CORPORATE SOURCE: Department of Chemistry, University of California,  
Davis, Davis, CA, 95616, USA

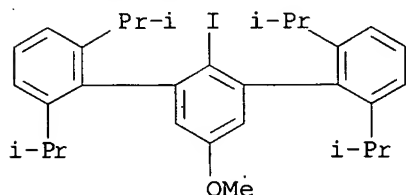
SOURCE: Journal of the American Chemical Society (2007),  
129(51), 16197-16208

PUBLISHER:  
DOCUMENT TYPE:  
LANGUAGE:

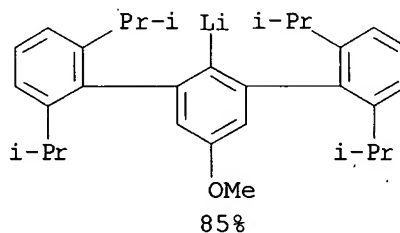
American Chemical Society  
Journal  
English

AB A series of sym. divalent Sn(II) hydrides of the general form  $[(4-X-Ar')Sn(\mu-H)]_2$  ( $4-X-Ar' = C_6H_2-4-X-2,6-(C_6H_3-2,6-iPr_2)_2$ ;  $X = H, MeO, tBu$ , and  $SiMe_3$ ; 2, 6, 10, and 14), along with the more hindered asym. tin hydride  $(3,5-iPr_2-Ar^*)SnSn(H)_2(3,5-iPr_2-Ar^*)$  (16) ( $3,5-iPr_2-Ar^* = 3,5-iPr_2-C_6H_2-2,6-(C_6H_2-2,4,6-iPr_3)_2$ ), have been isolated and characterized. They were prepared either by direct reduction of the corresponding aryltin(II) chloride precursors,  $ArSnCl$ , with  $LiBH_4$  or  $iBu_2AlH$  (DIBAL), or via a transmetalation reaction between an aryltin(II) amide,  $ArSnNMe_2$ , and  $BH_3 \cdot THF$ . Compds. 2, 6, 10, and 14 were obtained as orange solids and have centrosym. dimeric structures in the solid state with long  $Sn \cdots Sn$  sepns. of 3.05 to 3.13 Å. The more hindered tin(II) hydride 16 crystallized as a deep-blue solid with an unusual, formally mixed-valent structure wherein a long Sn-Sn bond is present [ $Sn-Sn = 2.9157(10)$  Å] and two hydrogen atoms are bound to one of the tin atoms. The Sn-H hydrogen atoms in 16 could not be located by x-ray crystallog., but complementary Moessbauer studies established the presence of divalent and tetravalent tin centers in 16. Spectroscopic studies (IR, UV-vis, and NMR) show that, in solution, compds. 2, 6, 10, and 14 are predominantly dimeric with Sn-H-Sn bridges. In contrast, the more hindered hydrides 16 and previously reported  $(Ar^*SnH)_2$  (17) ( $Ar^* = C_6H_3-2,6-(C_6H_2-2,4,6-iPr_3)_2$ ) adopt primarily the unsym. structure  $ArSnSn(H)_2Ar$  in solution. Detailed theor. calcns. have been performed which include calculated UV-vis and IR spectra of various possible isomers of the reported hydrides and relevant model species. These showed that increased steric hindrance favors the asym. form  $ArSnSn(H)_2Ar$  relative to the centrosym. isomer  $[ArSn(\mu-H)]_2$  as a result of the widening of the interligand angles at tin, which lowers steric repulsion between the terphenyl ligands.

RX(4) OF 42

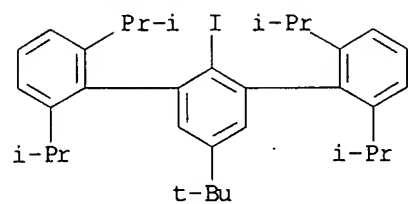


$BuLi$ , Hexane  $\rightarrow$

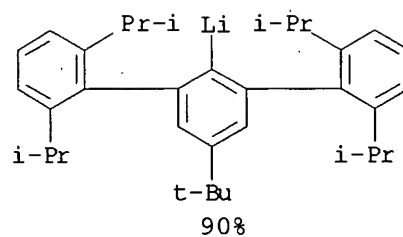


CON: 15 minutes, room temperature

RX(8) OF 42

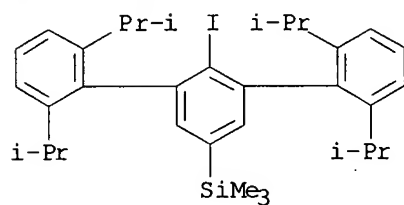


BuLi, Hexane →

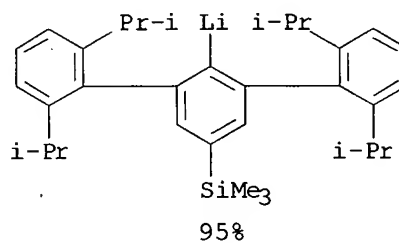


CON: 2 days, 0 deg C → room temperature

RX(12) OF 42

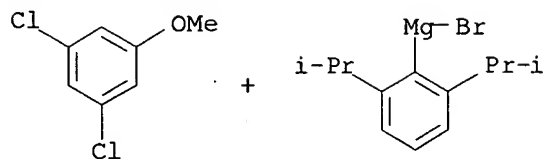


BuLi, Hexane →

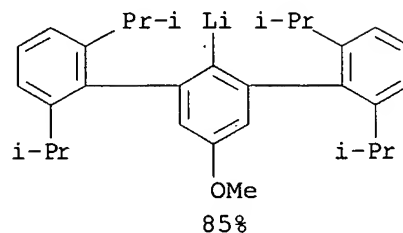


CON: 12 hours, 0 deg C → room temperature

RX(20) OF 42 - 2 STEPS



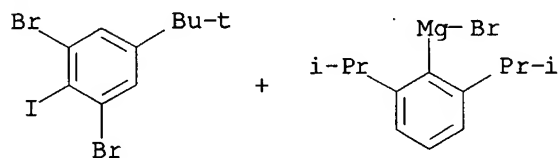
1.1. BuLi, THF,  
Hexane  
1.3. I2  
2. BuLi, Hexane



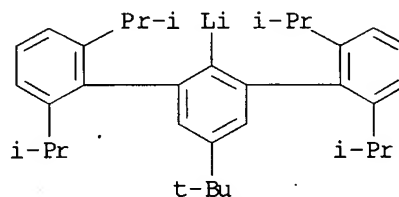
85%

CON: STEP(1.1) 3 hours, -78 deg C  
STEP(1.2) 3 hours, -78 deg C; 2 hours, reflux  
STEP(1.3) 80 minutes, 0 deg C  
STEP(2) 15 minutes, room temperature

RX(23) OF 42 - 2 STEPS



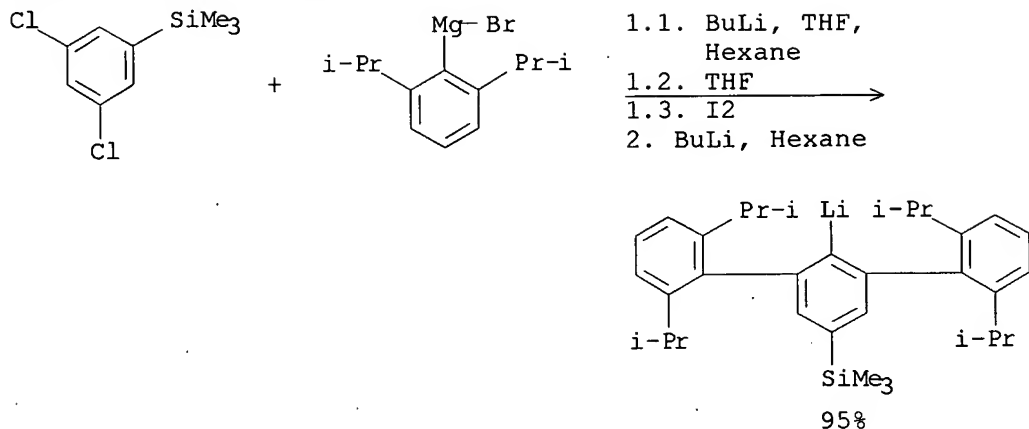
1.1. THF  
1.2. I2  
2. BuLi, Hexane



90%

CON: STEP(1.1) 0 deg C; 20 hours, reflux  
STEP(1.2) 16 hours, 0 deg C -> reflux  
STEP(2) 2 days, 0 deg C -> room temperature

RX(26) OF 42 - 2 STEPS



CON: STEP(1.1) 2 hours, -78 deg C  
STEP(1.2) overnight, -78 deg C -> room temperature; 2 hours, reflux  
STEP(1.3) 2 hours, 0 deg C -> reflux  
STEP(2) 12 hours, 0 deg C -> room temperature

REFERENCE COUNT: 78 THERE ARE 78 CITED REFERENCES AVAILABLE FOR THIS RECORD. ALL CITATIONS AVAILABLE IN THE RE FORMAT

L4 ANSWER 2 OF 50 CASREACT COPYRIGHT 2008 ACS on STN

ACCESSION NUMBER: 147:502456 CASREACT

TITLE: New bis(silyl)cyclopentadienylidoniobium and -tantalum complexes: X-ray crystal structures of  $[\text{NbCp}^*\text{Cl}_4]$  and  $[\text{NbCp}^*\text{Cl}_4(\text{CNAr})]$  [ $\text{Cp}^* = \eta^5\text{-C}_5\text{H}_3(\text{SiClMe}_2)(\text{SiMe}_3)$ ;  $\text{Ar} = 2,6\text{-Me}_2\text{C}_6\text{H}_3$ ]

AUTHOR(S): Gomez, Manuel; Gomez-Sal, Pilar; Hernandez, Jose Manuel

CORPORATE SOURCE: Departamento de Quimica Inorganica, Universidad de Alcala de Henares, Alcala de Henares, 28871, Spain

SOURCE: European Journal of Inorganic Chemistry (2006), (24), 5106-5114

CODEN: EJICFO; ISSN: 1434-1948

PUBLISHER: Wiley-VCH Verlag GmbH & Co. KGaA

DOCUMENT TYPE: Journal

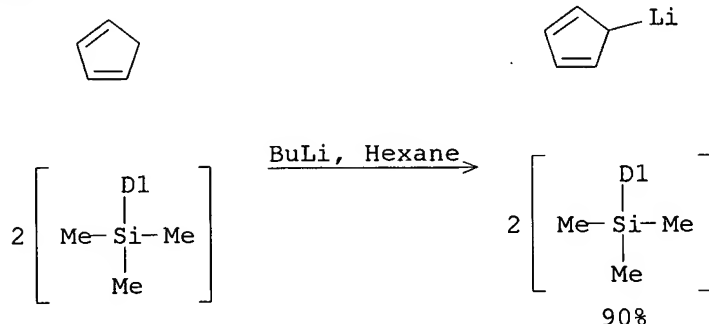
LANGUAGE: English

AB The [bis(silyl)cyclopentadienylidoniobium and -tantalum complexes  $[\text{MCp}^*\text{Cl}_4]$  [ $\text{Cp}^* = \eta^5\text{-C}_5\text{H}_3(\text{SiClMe}_2)(\text{SiMe}_3)$ ;  $\text{M} = \text{Nb}$  3,  $\text{Ta}$  4] were synthesized by reaction of the pentachlorides  $\text{MCl}_5$  with  $\text{C}_5\text{H}_3(\text{SiClMe}_2)(\text{SiMe}_3)_2$  (1). Although the Lewis acidity of tetrachloro complexes 3 and 4 is lower than that of the pentahalides, two adducts  $[\text{M}\{\eta^5\text{-C}_5\text{H}_3(\text{SiClMe}_2)(\text{SiMe}_3)\}\text{Cl}_4(\text{CNAr})]$  ( $\text{Ar} = 2,6\text{-Me}_2\text{C}_6\text{H}_3$ ;  $\text{M} = \text{Nb}$  5,  $\text{Ta}$  6) have been isolated by reaction with  $\text{ArNC}$ . Complexes 3 and 4 react with tert-butylamine or lithium amides to afford the dichloroimido and amidochloroimido complexes  $[\text{M}\{\eta^5\text{-C}_5\text{H}_3(\text{SiClMe}_2)(\text{SiMe}_3)\}\text{Cl}_2(\text{NR})]$  ( $\text{R} = \text{tBu}$ ,  $\text{M} = \text{Nb}$  7,  $\text{Ta}$  8;  $\text{R} = \text{Me}$ ,  $\text{M} = \text{Nb}$  9) and  $[\text{Ta}\{\eta^5\text{-C}_5\text{H}_3(\text{SiClMe}_2)(\text{SiMe}_3)\}\text{Cl}(\text{NHTBu})(\text{NTBu})]$  (10), resp. In addition, 7 and 8 can be prepared by treatment of the pentachlorides with  $[\text{C}_5\text{H}_3(\text{SiMe}_2\text{NHTBu})(\text{SiMe}_3)_2]$  (2) by elimination of 1. The tetrachloro compound 3 reacts with four equivalent of  $\text{tBuNH}_2$  to give the constrained-geometry derivative  $[\text{Nb}\{\eta^5\text{-C}_5\text{H}_3(\text{SiMe}_2\text{NTBu}-\kappa\text{N})(\text{SiMe}_3)\}\text{Cl}(\text{NTBu})]$  (11), whereas the treatment of toluene solns. of 3 and 4 with  $\text{H}_2\text{NCH}_2\text{CH}_2\text{NH}_2$  in the presence of triethylamine leads to the trichloro complexes  $[\text{M}\{\eta^5\text{-C}_5\text{H}_3(\text{SiMe}_2\text{NCH}_2\text{CH}_2\text{NH}_2-\kappa^2\text{N,N})(\text{SiMe}_3)\}\text{Cl}_3]$  ( $\text{M} = \text{Nb}$ ,  $\text{Ta}$ ). All the reported complexes were



studied by IR and NMR spectroscopy and the mol. structures of complexes 3 and 5 were determined by x-ray diffraction methods.

RX(14) OF 56



CON: 20 hours, 0 deg C -> room temperature

REFERENCE COUNT: 45 THERE ARE 45 CITED REFERENCES AVAILABLE FOR THIS RECORD. ALL CITATIONS AVAILABLE IN THE RE FORMAT

L4 ANSWER 3 OF 50 CASREACT COPYRIGHT 2008 ACS on STN

ACCESSION NUMBER: 146:500547 CASREACT

TITLE: 2- $\alpha$ -Hydroxyalkyl- and 2,7-Di( $\alpha$ -hydroxyalkyl)-1,8-bis(dimethylamino)naphthalenes: Stabilization of Nonconventional In/Out Conformers of "Proton Sponges" via N $\cdots$ H-O Intramolecular Hydrogen Bonding. A Remarkable Kind of Tandem Nitrogen Inversion

AUTHOR(S): Pozharskii, Alexander F.; Degtyarev, Alexander V.; Ryabtsova, Oksana V.; Ozeryanskii, Valery A.; Kletskii, Mikhail E.; Starikova, Zoya A.; Sobczyk, Lucjan; Filarowski, Alexander

CORPORATE SOURCE: Department of Organic Chemistry, Southern Federal University, Rostov-on-Don, 344090, Russia

SOURCE: Journal of Organic Chemistry (2007), 72(8), 3006-3019  
CODEN: JOCEAH; ISSN: 0022-3263

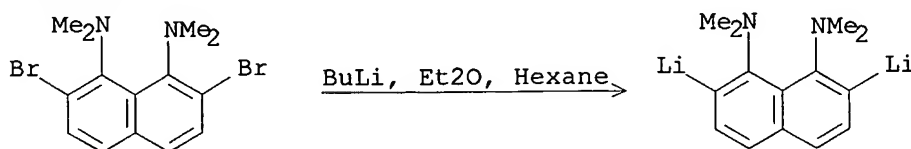
PUBLISHER: American Chemical Society

DOCUMENT TYPE: Journal

LANGUAGE: English

AB A regular set of 2-( $\alpha$ -hydroxymethyl)- and 2,7-di( $\alpha$ -hydroxymethyl)-1,8-bis(dimethylamino)naphthalenes has been prepared Their X-ray, NMR, and IR studies have demonstrated that in tertiary mono-alcs. the orientation of free nitrogen electron pairs in crystals and solution corresponds to nonconventional in/out conformers stabilized by O-H $\cdots$ N intramol. hydrogen bonding. For tertiary 2,7-dialcs., the superimposed equilibrating in/out-out/in nitrogen invertomers are observed in solution Unlike this, primary and secondary mono- and dialcs. commonly exist in the in/in form, which is typical for the parent proton sponge and the majority of its derivs.

RX(1) OF 41



CON: STAGE(1) 10 minutes, -20 deg C; -20 deg C

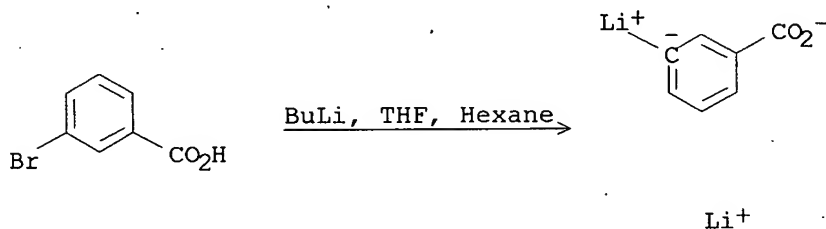
REFERENCE COUNT: 24 THERE ARE 24 CITED REFERENCES AVAILABLE FOR THIS RECORD. ALL CITATIONS AVAILABLE IN THE RE FORMAT

L4 ANSWER 4 OF 50 CASREACT COPYRIGHT 2008 ACS on STN

ACCESSION NUMBER: 146:379649 CASREACT  
TITLE: Synthesis and  $^{125}\text{I}$  labeling of N-succinimidyl-3-(tri-n-butylstannyl)benzoate  
AUTHOR(S): Liu, Zhenfeng; Wang, Yongxian; Zhou, Wei; Wang, Lihua; Xia, Jiaoyun; Yin, Duanzhi  
CORPORATE SOURCE: Shanghai Institute of Applied Physics, Chinese Academy of Sciences, Shanghai, 201800, Peop. Rep. China  
SOURCE: Tongweisu (2005), 18(3), 148-152  
CODEN: TONGEM; ISSN: 1000-7512  
PUBLISHER: Yuanzineng Chubanshe  
DOCUMENT TYPE: Journal  
LANGUAGE: Chinese

AB N-Succinimidyl-3-(tri-n-butylstannyl) benzoate (ATE) and N-succinimidyl-3-iodobenzoate (SIB) were synthesized. The structures of ATE and SIB were confirmed with  $^1\text{H}$ NMR, MS, and IR. The yields of ATE and SIB were 45.4% and 71.4%, resp. ATE was labeled with  $^{125}\text{I}$ . The labeling yield was 93.0% and radiochem. purity was over 98.0%. The synthesis and the labeling of ATE has a important value for indirect label of radiopharmaceuticals.

RX(1) OF 11



CON: STAGE(1) reflux; reflux  $\rightarrow$  -90 deg C; -90 deg C; 0.5 hours, -90 deg C; -90 deg C  $\rightarrow$  -80 deg C; 0.5 hours, -80 deg C

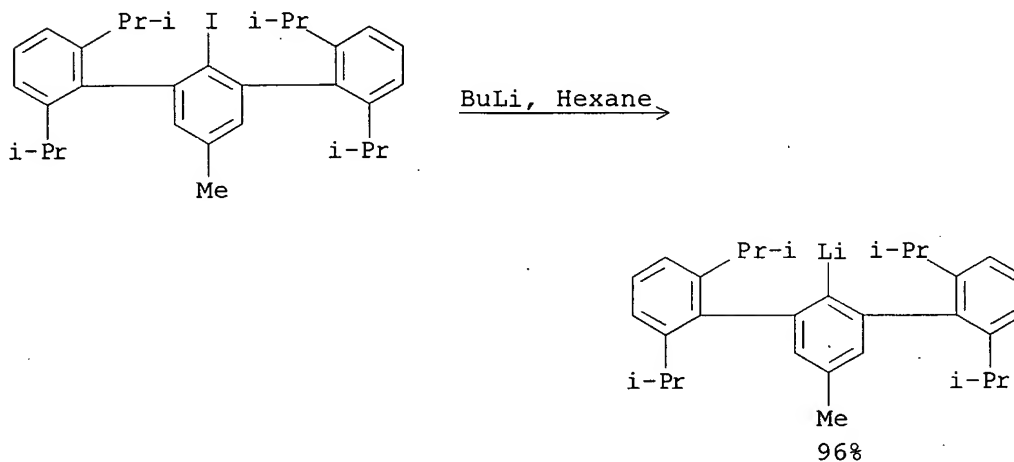
L4 ANSWER 5 OF 50 CASREACT COPYRIGHT 2008 ACS on STN

ACCESSION NUMBER: 145:103758 CASREACT  
TITLE: ipso-and para-Functionalization of meta-terphenyl ligands with substituted methyl groups: Unusual head-to-tail coupling of terphenyl moieties  
AUTHOR(S): Stanciu, Corneliu; Fox, Alexander R.; Richards, Anne F.; Fettingner, James C.; Power, Philip P.  
CORPORATE SOURCE: Department of Chemistry, University of California, Davis, CA, 95616, USA  
SOURCE: Journal of Organometallic Chemistry (2006), 691(11), 2546-2553  
CODEN: JORCAI; ISSN: 0022-328X  
PUBLISHER: Elsevier B.V.  
DOCUMENT TYPE: Journal  
LANGUAGE: English

AB The synthesis and characterization of several ipso-functionalized derivs. of the bulky terphenyl group  $\text{C}_6\text{H}_3\text{-2,6}(\text{C}_6\text{H}_3\text{-2',6'-Pri}_2)_2$  ( $\text{Ar}'$ ) are described. These include the primary alc.  $\text{Ar}'\text{CH}_2\text{OH}$  (1), the bromo derivative  $\text{Ar}'\text{CH}_2\text{Br}$  (2), and the terphenyl formate  $\text{Ar}'\text{CH}_2\text{OC(O)H}$  (3). The alc. 1 was obtained by treatment of  $\text{LiAr}'$  with formaldehyde, and 1 was readily converted to the bromo derivative 2 using  $\text{HBr}$ . The reaction of 1 with formic acid afforded 3 in good yield. Attempts to form the Grignard derivative of 1,

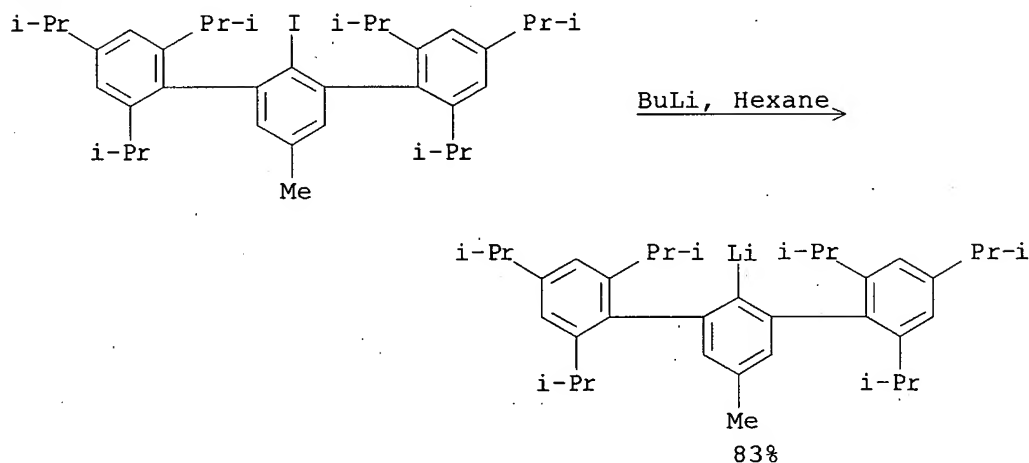
i.e.,  $\text{Ar}'\text{CH}_2\text{MgBr}$ , resulted in a head-to-tail reaction of the terphenyl benzyl units to yield an unusual coupled product 4. An approach to the avoidance of this coupling involved the synthesis of the terphenyl derivs. 1-I-C<sub>6</sub>H<sub>2</sub>-2,6(C<sub>6</sub>H<sub>3</sub>-2',6'-Pri<sub>2</sub>)<sub>2</sub>-4-Me (5) and 1-I-C<sub>6</sub>H<sub>2</sub>-2,6(C<sub>6</sub>H<sub>2</sub>-2',4',6'-Pri<sub>3</sub>)<sub>2</sub>-4-Me (6), bearing Me groups in the para positions of the central aryl ring, which could be prepared in good yield, and converted to their resp. lithium salts 7 and 8 without complication. The compds. were characterized by <sup>1</sup>H and <sup>13</sup>C NMR spectroscopy, IR spectroscopy (1) and x-ray crystallog. (2, 4, 5 and 6).

RX(7) OF 18



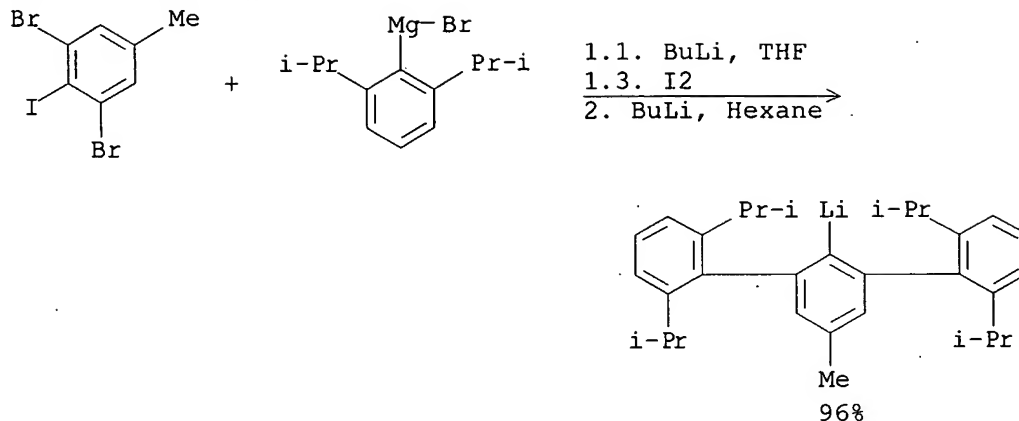
CON: 6 hours, 0 deg C -> room temperature

RX(8) OF 18



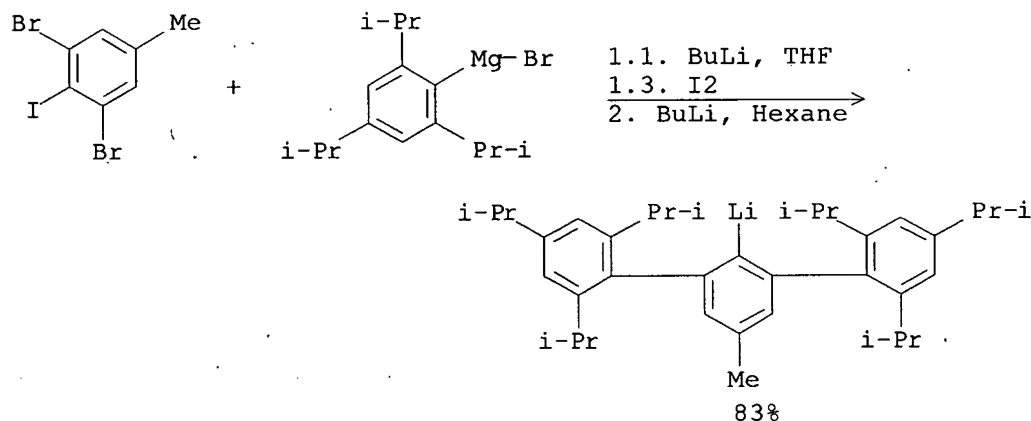
CON: 6 hours, 0 deg C -> room temperature

RX(13) OF 18 - 2 STEPS



CON: STEP(1.1) 20 minutes, -78 deg C  
STEP(1.2) overnight, room temperature; 2 hours, reflux  
STEP(1.3) 6 hours, room temperature  
STEP(2) 6 hours, 0 deg C -> room temperature

RX(14) OF 18 - 2 STEPS



CON: STEP(1.1) 20 minutes, -78 deg C  
STEP(1.2) overnight, room temperature; 2 hours, reflux  
STEP(1.3) 6 hours, room temperature  
STEP(2) 6 hours, 0 deg C -> room temperature

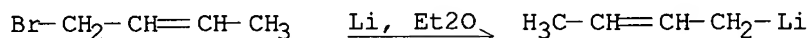
REFERENCE COUNT: 44 THERE ARE 44 CITED REFERENCES AVAILABLE FOR THIS RECORD. ALL CITATIONS AVAILABLE IN THE RE FORMAT

L4 ANSWER 6 OF 50 CASREACT COPYRIGHT 2008 ACS on STN  
ACCESSION NUMBER: 144:23019 CASREACT  
TITLE: Synthesis and characteristics of Megastigmatrienone  
AUTHOR(S): Wang, Jian-lin; Yang, Shao-long; Xu, Yan-mei; Zhao, Qing-hua; Qian, Lan  
CORPORATE SOURCE: Materials and Chemical Engineering College of Zhengzhou, University of Light Industry, Zhengzhou, 450002, Peop. Rep. China  
SOURCE: Guangpuxue Yu Guangpu Fenxi (2005), 25(3), 467-469  
CODEN: GYGFD; ISSN: 1000-0593  
PUBLISHER: Beijing Daxue Chubanshe  
DOCUMENT TYPE: Journal

LANGUAGE: Chinese

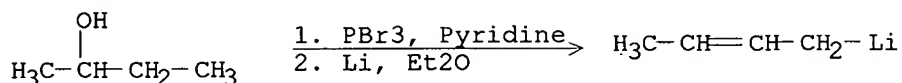
AB Megastigmatrienone was synthesized from oxyisophorone and crotonaldehyde, first protected the carbonyl group with ethylene glycol, then converted the crotonaldehyde to the corresponding bromide and lithium salt, further condensation of the two obtained intermediate and dehydration to form the title product. This route has the advantage of easily available starting material, mild reaction condition, high yield. All the product structures were characterized by IR, <sup>1</sup>H NMR, and MS, or refractive index.

RX(4) OF 13



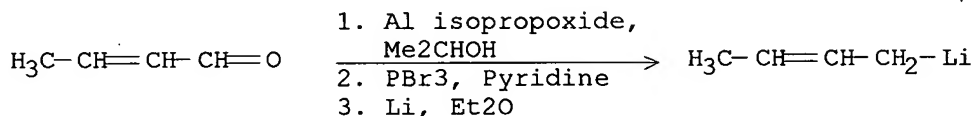
CON: 30 minutes, reflux

RX(8) OF 13 - 2 STEPS



CON: STEP(1.1) room temperature -> 10 deg C; 15 deg C; 1 hour,  
10 deg C  
STEP(2) 30 minutes, reflux

RX(11) OF 13 - 3 STEPS



CON: STEP(1) 36 hours, reflux  
STEP(2.1) room temperature -> 10 deg C; 15 deg C; 1 hour,  
10 deg C  
STEP(3) 30 minutes, reflux

L4 ANSWER 7 OF 50 CASREACT COPYRIGHT 2008 ACS on STN

ACCESSION NUMBER: 143:306390 CASREACT

TITLE: Novel [3]ferrocenophanes: Syntheses, redox properties and molecular structures of [Fe{(η<sup>5</sup>-C<sub>5</sub>H<sub>4</sub>)CMe<sub>2</sub>}<sub>2</sub>PR] (R = Ph, Cy)

AUTHOR(S): Hoecher, Thomas; Cinquantini, Arnaldo; Zanello, Piero; Hey-Hawkins, Evamarie

CORPORATE SOURCE: Institut fuer Anorganische Chemie, Universitaet Leipzig, Leipzig, D-04103, Germany

SOURCE: Polyhedron (2005), 24(11), 1340-1346  
CODEN: PLYHDE; ISSN: 0277-5387

PUBLISHER: Elsevier B.V.

DOCUMENT TYPE: Journal

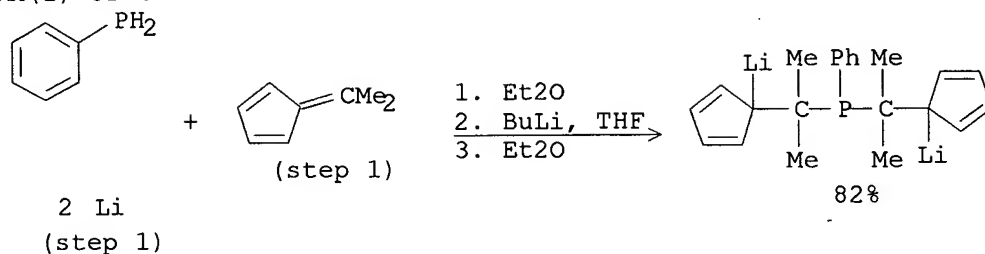
LANGUAGE: English

AB 6,6-Dimethylfulvene reacts with Li<sub>2</sub>PR to give Li<sub>2</sub>[(η<sup>5</sup>-C<sub>5</sub>H<sub>4</sub>)CMe<sub>2</sub>}<sub>2</sub>PR] [R = Ph (1), Cy (2)]. The ferrocenophanes [Fe{(η<sup>5</sup>-C<sub>5</sub>H<sub>4</sub>)CMe<sub>2</sub>}<sub>2</sub>PR] [R = Ph (3), Cy (4)] were obtained in good yield from the Li reagents 1 and 2 and FeCl<sub>2</sub>. Compds. 3 and 4 were characterized spectroscopically (<sup>1</sup>H, <sup>13</sup>C, <sup>31</sup>P, IR, MS), and by crystal structure determination. Electrochem. study shows that 3 undergoes a 1-electron oxidation in CH<sub>2</sub>Cl<sub>2</sub> solution, which is chemical

reversible on the short timescale of cyclic voltammetry, but in the longer time frame of macroelectrolysis the monocation [3]<sup>+</sup> forms a new species

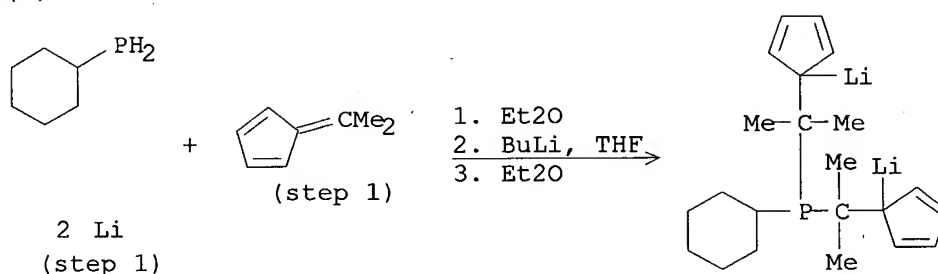
that the authors tentatively assign as  $[\text{Fe}(\eta^5\text{-C}_5\text{H}_4)\text{CMe}_2]_2\text{P}(\text{O})\text{Ph}]$ .

RX(1) OF 6



CON: STAGE(1) room temperature; 12 hours, room temperature  
STAGE(2) room temperature  
STAGE(3) room temperature; 12 hours, room temperature

RX(2) OF 6



NOTE: other products also detected, product contaminated with 30% monolithiated compd.

CON: STAGE(1) room temperature; 12 hours, room temperature  
STAGE(2) room temperature  
STAGE(3) room temperature; 12 hours, room temperature

REFERENCE COUNT: 45 THERE ARE 45 CITED REFERENCES AVAILABLE FOR THIS RECORD. ALL CITATIONS AVAILABLE IN THE RE FORMAT

L4 ANSWER 8 OF 50 CASREACT COPYRIGHT 2008 ACS on STN

ACCESSION NUMBER: 143:248504 CASREACT

TITLE: Method for producing alkyl lithium compounds and aryl lithium compounds by monitoring the reaction by means of ir-spectroscopy

INVENTOR(S): Weiss, Wilfried; Dawidowski, Dirk; Pleyer, Walter; Krueckel, Frank

PATENT ASSIGNEE(S): Chemetall G.m.b.H., Germany

SOURCE: PCT Int. Appl., 32 pp.

CODEN: PIXXD2

DOCUMENT TYPE: Patent

LANGUAGE: German

FAMILY ACC. NUM. COUNT: 1

PATENT INFORMATION:

PATENT NO.	KIND	DATE	APPLICATION NO.	DATE
WO 2005082911	A1	20050909	WO 2005-EP1954	20050224
W: AE, AG, AL, AM, AT, AU, AZ, BA, BB, BG, BR, BW, BY, BZ, CA, CH, CN, CO, CR, CU, CZ, DE, DK, DM, DZ, EC, EE, EG, ES, FI, GB, GD, GE, GH, GM, HR, HU, ID, IL, IN, IS, JP, KE, KG, KP, KR, KZ, LC, LK, LR, LS, LT, LU, LV, MA, MD, MG, MK, MN, MW, MX, MZ, NA, NI, NO, NZ, OM, PG, PH, PL, PT, RO, RU, SC, SD, SE, SG, SK, SL, SM, SY, TJ, TM, TN, TR, TT, TZ, UA, UG, US, UZ, VC, VN, YU, ZA, ZM, ZW				

RW: BW, GH, GM, KE, LS, MW, MZ, NA, SD, SL, SZ, TZ, UG, ZM, ZW, AM,  
 AZ, BY, KG, KZ, MD, RU, TJ, TM, AT, BE, BG, CH, CY, CZ, DE, DK,  
 EE, ES, FI, FR, GB, GR, HU, IE, IS, IT, LT, LU, MC, NL, PL, PT,  
 RO, SE, SI, SK, TR, BF, BJ, CF, CG, CI, CM, GA, GN, GQ, GW, ML,  
 MR, NE, SN, TD, TG

DE 102004009445 A1 20050929 DE 2004-10200400944520040227  
 EP 1723153 A1 20061122 EP 2005-733858 20050224

R: AT, BE, BG, CH, CY, CZ, DE, DK, EE, ES, FI, FR, GB, GR, HU, IE,  
 IS, IT, LI, LT, LU, MC, NL, PL, PT, RO, SE, SI, SK, TR

CN 1922192 A 20070228 CN 2005-80005827 20050224  
 IN 2006CN03106 A 20070608 IN 2006-CN3106 20060825  
 US 20070152354 A1 20070705 US 2006-589715 20061023

PRIORITY APPLN. INFO.:

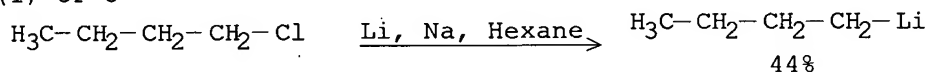
DE 2004-10200400944520040227  
 WO 2005-EP1954 20050224

OTHER SOURCE(S): MARPAT 143:248504

AB The invention relates to a method for producing alkyl lithium compds. and  
 aryl lithium compds. by reacting lithium metal with alkyl or aryl  
 halogenides in a solvent, the concentration of the alkyl/aryl halogenide and  
 the

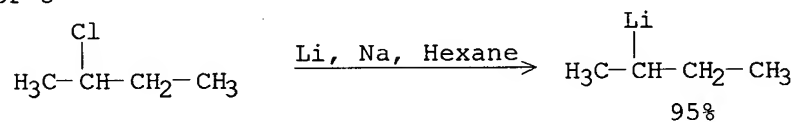
alkyl/aryl lithium compound being detected according to an in-line  
 measurement in the reactor by IR spectroscopy, and an exact  
 recognition of the end point of the dosing of the halogenide constituents  
 being carried out by evaluation of the IR measurement. Said  
 method enables an optimum reactive process and reaction yield. The  
 identification of the resp. concentration of the adduct and the product is a  
 reliable reactive process. The yield of the reaction is also optimized by  
 determining the end point of the halogenide dosing, as is the purity of the  
 product due to a lower concentration thereof during the reaction.

RX(1) OF 5



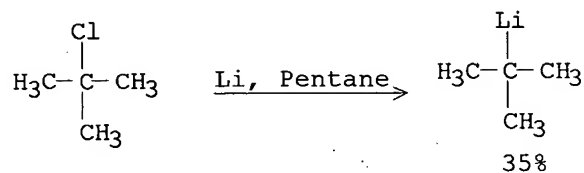
CON: 280 minutes, room temperature

RX(2) OF 5



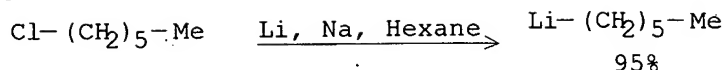
CON: 75 minutes, 40 deg C, 290 atm

RX(3) OF 5



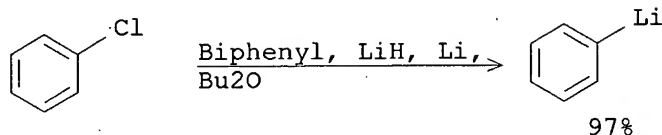
NOTE: tert-butyllithium mediated  
 CON: 144 minutes, room temperature

RX(4) OF 5



CON: 40 deg C, 290 atm

RX(5) OF 5



CON: 4 hours, 35 deg C

REFERENCE COUNT: 1 THERE ARE 1 CITED REFERENCES AVAILABLE FOR THIS RECORD. ALL CITATIONS AVAILABLE IN THE RE FORMAT

L4 ANSWER 9 OF 50 CASREACT COPYRIGHT 2008 ACS on STN

ACCESSION NUMBER: 142:373346 CASREACT

TITLE: Catalytic Asymmetric Cyano-Ethoxycarbonylation  
Reaction of Aldehydes using a YLi3Tris(binaphthoxide)  
(YLB) Complex: Mechanism and Roles of Achiral  
Additives

AUTHOR(S): Yamagiwa, Noriyuki; Tian, Jun; Matsunaga, Shigeki;  
Shibasaki, Masakatsu

CORPORATE SOURCE: Graduate School of Pharmaceutical Sciences, University  
of Tokyo, Tokyo, 113-0033, Japan

SOURCE: Journal of the American Chemical Society (2005),  
127(10), 3413-3422

CODEN: JACSAT; ISSN: 0002-7863

PUBLISHER: American Chemical Society

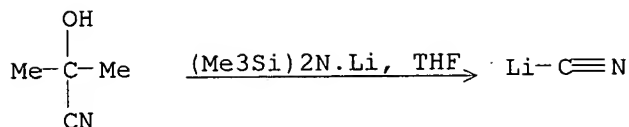
DOCUMENT TYPE: Journal

LANGUAGE: English

AB Full details of a catalytic asym. cyano-ethoxycarbonylation reaction promoted by a heterobimetallic YLi3tris(binaphthoxide) complex (YLB 1), especially mechanistic studies, are described. In the cyanation reaction of aldehydes with Et cyano-formate, three achiral additives, H<sub>2</sub>O, tris(2,6-dimethoxyphenyl)phosphine oxide (3a), and BuLi, were required to achieve high reactivity and enantioselectivity (up to >99% yield and up to 98% ee). The roles of achiral additives and the reaction pathway were investigated in detail. In situ IR anal. revealed that the initiation step to generate LiCN from H<sub>2</sub>O, BuLi, and Et cyano-formate is rather slow. On the basis of mechanistic studies of the initiation step to generate an active nucleophilic species, reaction conditions were optimized by using a catalytic amount of acetone cyanohydrin as an initiator. Under the optimized conditions, the induction period decreased and the reaction completed within 9 min using 5 mol % YLB at -78 °C. Catalyst loading was successfully reduced to 1 mol %. Kinetic expts. and evaluation of the substituent effects of phosphine oxide revealed that phosphine oxide had beneficial effects on both the reaction rate and the enantioselectivity. The putative active species as well as the catalytic cycle of the reaction are also discussed.



RX(2) OF 33



CON: 0.5 hours, 0 deg C

REFERENCE COUNT: 38 THERE ARE 38 CITED REFERENCES AVAILABLE FOR THIS RECORD. ALL CITATIONS AVAILABLE IN THE RE FORMAT

L4 ANSWER 10 OF 50 CASREACT COPYRIGHT 2008 ACS on STN

ACCESSION NUMBER: 142:280272 CASREACT

TITLE: Chiral Ansa Zirconocene Compounds with [Me<sub>2</sub>Si] and [Me<sub>4</sub>Si<sub>2</sub>] Bridges and with tert-Butyl Ring Substituents: Synthesis and Structural Characterization of the Racemo Complexes rac-[Me<sub>2</sub>Si(C<sub>5</sub>H<sub>2</sub>-2,4-But<sub>2</sub>)<sub>2</sub>]ZrCl<sub>2</sub> and rac-[Me<sub>4</sub>Si<sub>2</sub>(C<sub>5</sub>H<sub>2</sub>-2,4-But<sub>2</sub>)<sub>2</sub>]ZrCl<sub>2</sub>

AUTHOR(S): Zachmanoglou, Cary E.; Melnick, Jonathan G.; Bridgewater, Brian M.; Churchill, David G.; Parkin, Gerard

CORPORATE SOURCE: Department of Chemistry, Columbia University, New York, NY, 10027, USA

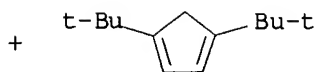
SOURCE: Organometallics (2005), 24(4), 603-611  
CODEN: ORGND7; ISSN: 0276-7333

PUBLISHER: American Chemical Society

DOCUMENT TYPE: Journal

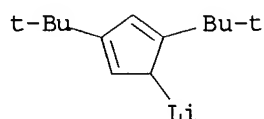
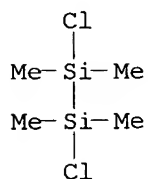
LANGUAGE: English

AB A series of chiral ansa zirconocene compds. that feature (i) [Me<sub>2</sub>Si] and [Me<sub>4</sub>Si<sub>2</sub>] bridges and (ii) two tert-Bu substituents on each cyclopentadienyl ring, namely, rac-[Me<sub>2</sub>Si(CpBut<sub>2</sub>)<sub>2</sub>]ZrCl<sub>2</sub>, rac-[Me<sub>2</sub>Si(CpBut<sub>2</sub>)<sub>2</sub>]Zr(Ph)Cl, rac-[Me<sub>2</sub>Si(CpBut<sub>2</sub>)<sub>2</sub>]ZrMe<sub>2</sub>, rac-[Me<sub>2</sub>Si(CpBut<sub>2</sub>)<sub>2</sub>]Zr(CO)<sub>2</sub>, rac-[Me<sub>4</sub>Si<sub>2</sub>(CpBut<sub>2</sub>)<sub>2</sub>]ZrCl<sub>2</sub>, and rac-[Me<sub>4</sub>Si<sub>2</sub>(CpBut<sub>2</sub>)<sub>2</sub>]Zr(CO)<sub>2</sub>, has been synthesized. In each case, the complex exists as the racemo isomer due to inter-ring steric interactions between the But substituents proximal to the ansa bridge that destabilize the meso isomer. Rac-[Me<sub>4</sub>Si<sub>2</sub>(CpBut<sub>2</sub>)<sub>2</sub>]ZrCl<sub>2</sub> is the first structurally characterized ansa zirconocene complex with a [Me<sub>4</sub>Si<sub>2</sub>] bridge that adopts a racemo geometry. IR spectroscopic studies on the dicarbonyl complexes (Cp<sub>1,3</sub>-But<sub>2</sub>)<sub>2</sub>Zr(CO)<sub>2</sub>, rac-[Me<sub>2</sub>Si(CpBut<sub>2</sub>)<sub>2</sub>]Zr(CO)<sub>2</sub>, and rac-[Me<sub>4</sub>Si<sub>2</sub>(CpBut<sub>2</sub>)<sub>2</sub>]Zr(CO)<sub>2</sub> demonstrate that the [Me<sub>2</sub>Si] and [Me<sub>4</sub>Si<sub>2</sub>] ansa bridges have opposite electronic influences in this system, with the [Me<sub>2</sub>Si] bridge exerting an electron-withdrawing effect and the [Me<sub>4</sub>Si<sub>2</sub>] bridge exerting an electron-donating effect. The [Me<sub>2</sub>Si] and [Me<sub>4</sub>Si<sub>2</sub>] bridges also exert a different influence with respect to olefin polymerization, with {rac-[Me<sub>2</sub>Si(CpBut<sub>2</sub>)<sub>2</sub>]ZrCl<sub>2</sub>/MAO} being a more active catalyst system for polymerization of ethylene than is either {(Cp<sub>1,3</sub>-But<sub>2</sub>)<sub>2</sub>ZrCl<sub>2</sub>/MAO} or {rac-[Me<sub>4</sub>Si<sub>2</sub>(CpBut<sub>2</sub>)<sub>2</sub>]ZrCl<sub>2</sub>/MAO}.

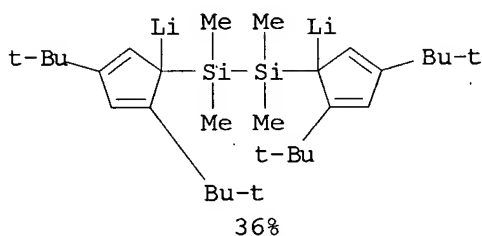
C[C@H]1C=C(C)C=C1C

t-Bu-C5H3(Li)-t-Bu  
 798

RX (9) OF 37



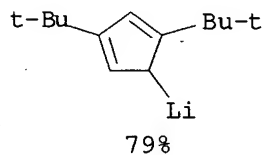
THE →



RX(11) OF 37 - 2 STEPS



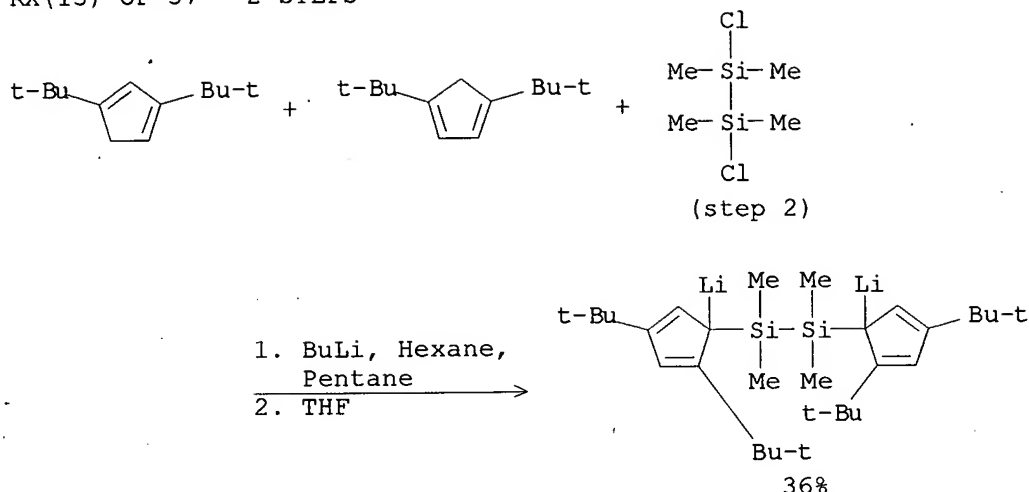
1. t-BuBr, NaH,  
Dibenzo-18-crown-6,  
THF
2. BuLi, Hexane,  
Pentane



CON: STEP(1.1) 12 hours, -78 deg C; 12 hours, 0 deg C; 12 hours, reflux

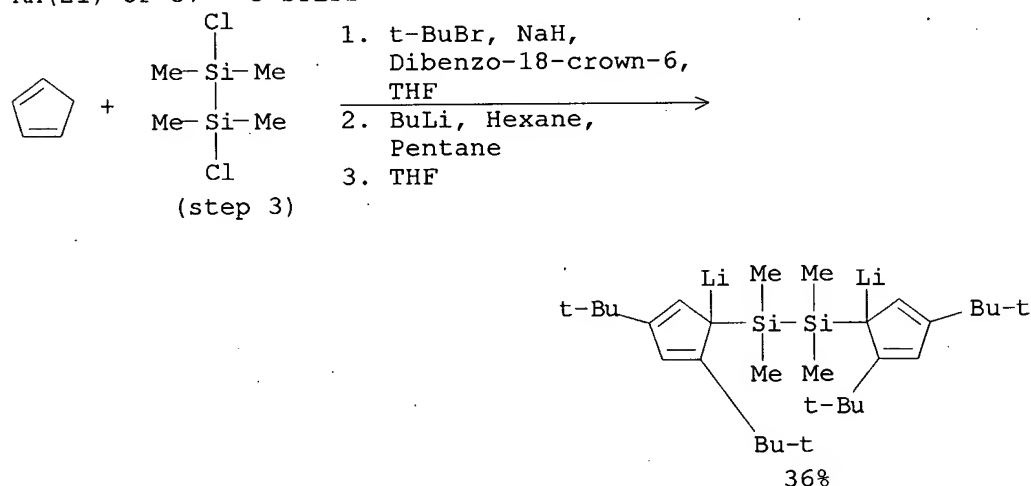
STEP(2) 12 hours, -78 deg C.-> room temperature

RX(13) OF 37 - 2 STEPS



CON: STEP(1) 12 hours, -78 deg C -> room temperature  
 STEP(2) 3 days, room temperature -> 95 deg C

RX(21) OF 37 - 3 STEPS



NOTE: 1) 57% overall

CON: STEP(1.1) 12 hours, -78 deg C; 12 hours, 0 deg C; 12 hours, reflux  
 STEP(2) 12 hours, -78 deg C -> room temperature  
 STEP(3) 3 days, room temperature -> 95 deg C

REFERENCE COUNT: 62 THERE ARE 62 CITED REFERENCES AVAILABLE FOR THIS RECORD. ALL CITATIONS AVAILABLE IN THE RE FORMAT

L4 ANSWER 11 OF 50 CASREACT COPYRIGHT 2008 ACS on STN

ACCESSION NUMBER: 141:424261 CASREACT

TITLE: Synthesis and characterization of zirconium and iron complexes containing substituted indenyl ligands: evaluation of steric and electronic parameters

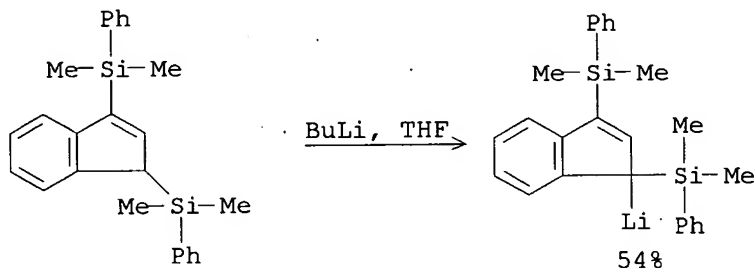
AUTHOR(S): Bradley, Christopher A.; Flores-Torres, Samuel; Lobkovsky, Emil; Abruna, Hector D.; Chirik, Paul J.

CORPORATE SOURCE: Department of Chemistry and Chemical Biology, Baker Laboratory, Cornell University, Ithaca, NY, 14853, USA

SOURCE: Organometallics (2004), 23(22), 5332-5346  
 CODEN: ORGND7; ISSN: 0276-7333  
 PUBLISHER: American Chemical Society  
 DOCUMENT TYPE: Journal  
 LANGUAGE: English

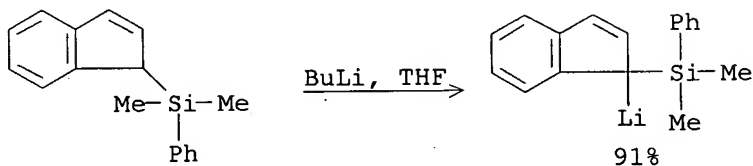
AB Evaluation of the steric and electronic influence of a family of silyl- and alkyl-substituted indenyl ligands on zirconium and iron centers has been accomplished by a combination of x-ray diffraction, IR spectroscopy, solution NMR dynamics, and electrochem. measurements. Three tetrasubstituted, bis-indenyl zirconocene dichloride complexes have been characterized by x-ray diffraction and adopt a gauche ligand conformation such that the interactions between tertiary substituents on adjacent rings are minimized. Similar solid state conformations were also observed in two of the corresponding iron compds. Evaluation of the electronic environment about each zirconium center was achieved by measurement of the CO stretching frequencies of the dicarbonyl derivs. Simple inductive effects govern the electronic properties of each zirconocene where silyl groups are relatively electron withdrawing and alkyl groups electron donating. For the most hindered zirconocene dicarbonyl derivs., population of three vibrationally distinct rotamers has been detected by IR spectroscopy. Independent assessment of these stereoelectronic parameters by variable-temperature NMR spectroscopy and electrochem. with the analogous series of iron complexes provided the same relative ordering of the indenyl ligands.

RX(28) OF 101



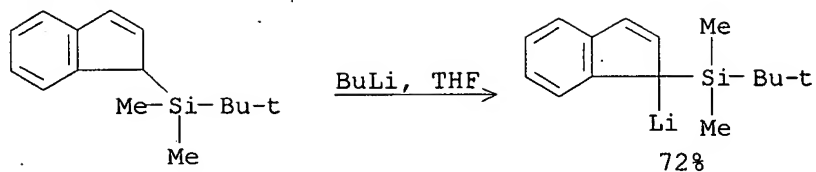
NOTE: cold well  
 CON: 30 minutes

RX(30) OF 101



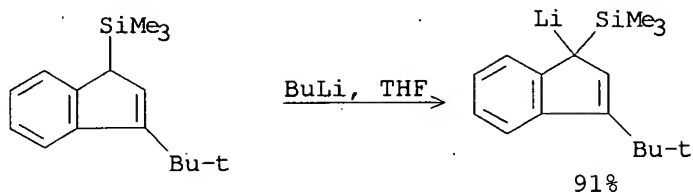
NOTE: cold well  
 CON: 30 minutes

RX(33) OF 101



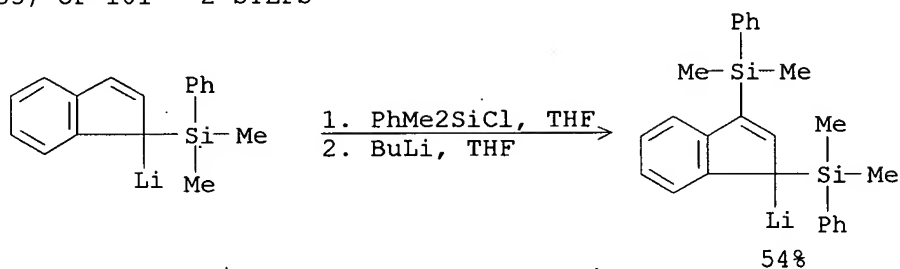
NOTE: cold well  
CON: 30 minutes

RX(36) OF 101



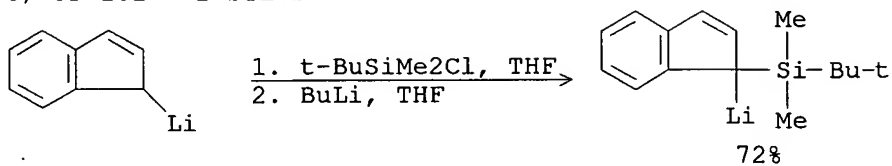
NOTE: cold well  
CON: 30 minutes

RX(55) OF 101 - 2 STEPS



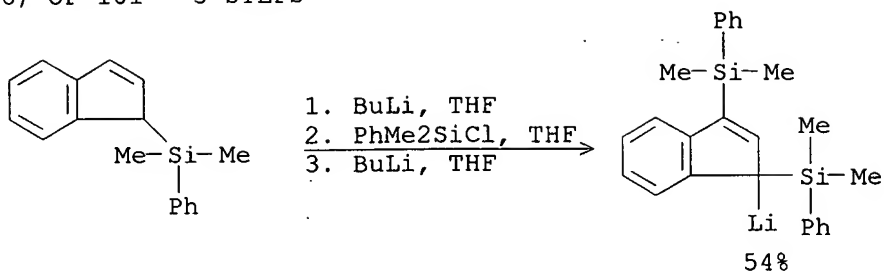
NOTE: 1) cold well, 2) cold well  
CON: STEP(1) 30 minutes  
STEP(2) 30 minutes

RX(56) OF 101 - 2 STEPS



NOTE: 1) cold well, 2) cold well  
CON: STEP(1) 30 minutes  
STEP(2) 30 minutes

RX(76) OF 101 - 3 STEPS



NOTE: 1) cold well, 2) cold well, 3) cold well

CON: STEP(1) 30 minutes

STEP(2) 30 minutes

STEP(3) 30 minutes

REFERENCE COUNT:

41

THERE ARE 41 CITED REFERENCES AVAILABLE FOR THIS RECORD. ALL CITATIONS AVAILABLE IN THE RE FORMAT

L4 ANSWER 12 OF 50 CASREACT COPYRIGHT 2008 ACS on STN

ACCESSION NUMBER: 140:357482 CASREACT

TITLE: Synthesis and reactivity of substituted cyclopentadienyl rhodium(I) and (III) complexes

AUTHOR(S): Xu, You-Feng; Shen, Yan; Pang, Zhen

CORPORATE SOURCE: Department of Chemistry, Fudan University, Shanghai, 200433, Peop. Rep. China

SOURCE: Journal of Organometallic Chemistry (2004), 689(4), 823-832

CODEN: JORCAI; ISSN: 0022-328X

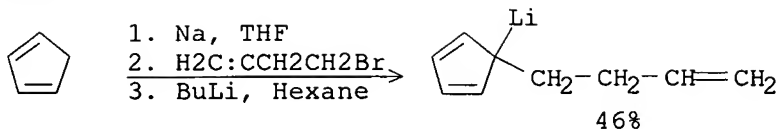
PUBLISHER: Elsevier Science B.V.

DOCUMENT TYPE: Journal

LANGUAGE: English

AB New cyclopentadienyl derivs. of Rh COD complexes [Cp\**C*5H4COOCH2CH:CH2 (1); *C*5H4CH2CH2CH:CH2 (2); *C*5H(*i*-Pr)<sub>4</sub> (3)] and carbonyl complex [Cp\**C*5H(*i*-Pr)<sub>4</sub> (4)] were synthesized from [RhCl(COD)]<sub>2</sub> and [RhCl(CO)<sub>2</sub>]<sub>2</sub>. 1, 2 And 3 oxidized by I gave I bridged dimers 5, 6 and 7, resp. Tri-Ph phosphine, CO and CS<sub>2</sub> mols. broke down the I bridged structure easily and produced monomer products Cp\*RhI<sub>2</sub>L [Cp\**C*5H4COOCH2CH:CH2, L = CS<sub>2</sub> (8); L = PPh<sub>3</sub> (9). Cp\**C*5H(*i*-Pr)<sub>4</sub>, L = CO (10)]. All of these new compds. were characterized by elemental anal., <sup>1</sup>H NMR, IR, UV-visible and mass spectroscopy. The crystal structure of 1 was solved in the triclinic space group P1 with one mol. in the unit cell, the dimensions of which are *a* = 7.082(9) Å, *b* = 8.392(3) Å, *c* 13.889(5) Å, α 101.19(3), β 99.06(6), γ 105.11(5)°, and *V* = 763(1) Å<sup>3</sup>. The crystal structure of 3 was solved in the orthorhombic space group Pn21α with four mols. in the unit cell, the dimensions of which are *a* = 9.748(3) Å, *b* = 16.054(5) Å, *c* 14.816(4) Å and *V* = 2319(1) Å<sup>3</sup>. Least squares refinement leads to values for the conventional R1 of 0.0251 for 1 and 0.0558 for 3, resp. Compared to that in 1, a shorter metal-ligand bond length in 3 was observed and this is attributed to the rich electron d. on Rh(I) metal center piled up by the *C*5H(*i*-Pr)<sub>4</sub> ligand.

RX(11) OF 27



CON: STAGE(1) 0 deg C; 0 deg C -> -40 deg C  
STAGE(2) 2 hours, -40 deg C  
STAGE(3) 40 minutes, -45 deg C; 180 minutes, -45 deg C

REFERENCE COUNT: 38 THERE ARE 38 CITED REFERENCES AVAILABLE FOR THIS RECORD. ALL CITATIONS AVAILABLE IN THE RE FORMAT

L4 ANSWER 13 OF 50 CASREACT COPYRIGHT 2008 ACS on STN

ACCESSION NUMBER: 140:235815 CASREACT

TITLE: Linear Hybrid Aminoborane/Phosphinoborane Chains: Synthesis, Proton-Hydride Interactions, and Thermolysis Behavior

AUTHOR(S): Jaska, Cory A.; Lough, Alan J.; Manners, Ian  
CORPORATE SOURCE: Department of Chemistry, University of Toronto, Toronto, ON, M5S 3H6, Can.

SOURCE: Inorganic Chemistry (2004), 43(3), 1090-1099  
CODEN: INOCAJ; ISSN: 0020-1669

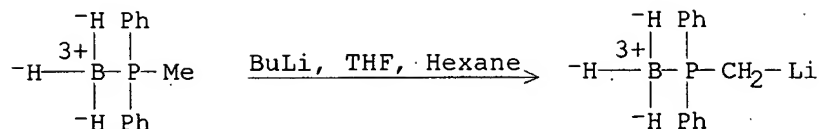
PUBLISHER: American Chemical Society

DOCUMENT TYPE: Journal

LANGUAGE: English

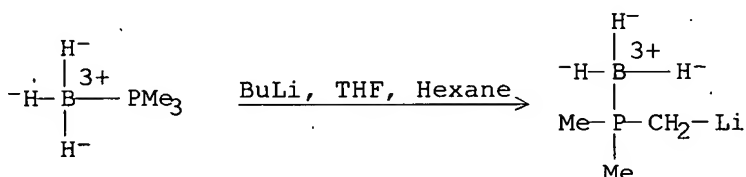
AB The reaction of the lithiated phosphine-borane adducts Li[PPhR·BH<sub>3</sub>] or Li[CH<sub>2</sub>-PR<sub>2</sub>·BH<sub>3</sub>] with Me<sub>2</sub>NH·BH<sub>2</sub>Cl afforded the hybrid linear species Me<sub>2</sub>NH-BH<sub>2</sub>-PPhR-BH<sub>3</sub> (1, R = Ph; 2, R = H) or Me<sub>2</sub>NH-BH<sub>2</sub>-CH<sub>2</sub>-PR<sub>2</sub>-BH<sub>3</sub> (3, R = Ph; 4, R = Me). Single-crystal x-ray diffraction studies on 1 and 3, the first for linear hybrid aminoborane/phosphinoborane adducts, confirmed the expected four-coordinate N-B-P-B and N-B-C-P-B frameworks. In addition, interactions between the protic N-H and hydridic B-H hydrogen atoms resulted in short intermol. H···H contacts for 1, whereas 3 was found to possess an exceptionally short intramol. H···H distance of 1.95 Å. Solution and solid state IR studies on 3 and 4 also suggest that these dihydrogen interactions were maintained even in dilute solution. Hydrogen bond strengths in the range of 7.9 to 10.9 kJ mol<sup>-1</sup> indicate the presence of a relatively weak interaction. The thermal and catalytic dehydrocoupling reactivities of 1-4 were also investigated. Chain cleavage reactions were observed for 1 and 2 upon thermolysis at 130° to afford species such as Me<sub>2</sub>NH·BH<sub>3</sub>, [Me<sub>2</sub>N-BH<sub>2</sub>]<sub>2</sub>, PhPRH·BH<sub>3</sub> (R = Ph, H), PhPRH (R = Ph, H), Ph<sub>2</sub>PH-BH<sub>2</sub>-PPh<sub>2</sub>-BH<sub>3</sub>, and also the low mol. weight polyphosphinoborane [PhPH-BH<sub>2</sub>]<sub>n</sub> (Mw .apprx. 5000). Similar products were observed for the attempted catalytic dehydrocoupling reactions but under milder reaction conditions (50°). Thermolysis of 3 at 130° yielded the six-membered ring [BH<sub>2</sub>-CH<sub>2</sub>-PPh<sub>2</sub>]<sub>2</sub> (5), which presumably results from the dissociation of Me<sub>2</sub>NH·BH<sub>3</sub> from 3. Thermolysis of 4 at 90° afforded Me<sub>2</sub>NH·BH<sub>3</sub> and Me<sub>3</sub>P·BH<sub>3</sub>, in addition to a product tentatively assigned as [BH<sub>2</sub>-CH<sub>2</sub>-PMe<sub>2</sub>]<sub>2</sub> (6).

RX(10) OF 32



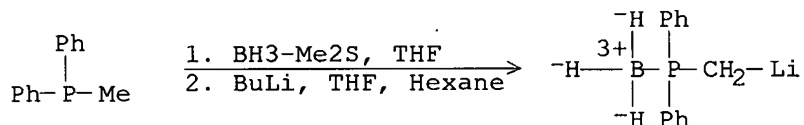
CON: STAGE(1) 30 minutes, 0 deg C; 90 minutes, 25 deg C

RX(11) OF 32



CON: STAGE(1) 30 minutes, 0 deg C; 90 minutes, 25 deg C

RX(13) OF 32 - 2 STEPS



CON: STEP(1) overnight, -30 deg C -> 25 deg C  
STEP(2.1) 30 minutes, 0 deg C; 90 minutes, 25 deg C

REFERENCE COUNT: 50 THERE ARE 50 CITED REFERENCES AVAILABLE FOR THIS RECORD. ALL CITATIONS AVAILABLE IN THE RE FORMAT

L4 ANSWER 14 OF 50 CASREACT COPYRIGHT 2008 ACS on STN

ACCESSION NUMBER: 139:230825 CASREACT

TITLE: Bis(phosphino)borates: A New Family of Monoanionic Chelating Phosphine Ligands

AUTHOR(S): Thomas, J. Christopher; Peters, Jonas C.

CORPORATE SOURCE: Division of Chemistry and Chemical Engineering Arnold and Mabel Beckman Laboratories of Chemical Synthesis, California Institute of Technology, Pasadena, CA, 91125, USA

SOURCE: Inorganic Chemistry (2003), 42(17), 5055-5073  
CODEN: INOCAJ; ISSN: 0020-1669

PUBLISHER: American Chemical Society

DOCUMENT TYPE: Journal

LANGUAGE: English

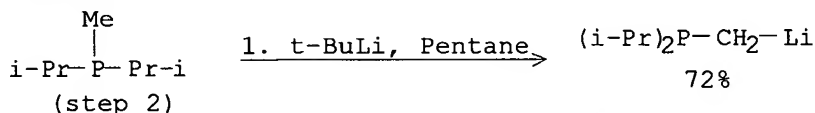
AB Preparation of a variety of diphosphines, bridged by neg. charged diaryl- or dialkylborato unit is described; their anionic dimethylplatinum and zwitterionic methyl(carbonyl)platinum complexes were prepared and characterized. Diarylchloroboranes R<sub>2</sub>BCl (10-15; R = Ph, p-MeC<sub>6</sub>H<sub>4</sub>, 3,5-Me<sub>2</sub>C<sub>6</sub>H<sub>4</sub>, 4-tBuC<sub>6</sub>H<sub>4</sub>, 4-MeOC<sub>6</sub>H<sub>4</sub>, 4-CF<sub>3</sub>C<sub>6</sub>H<sub>4</sub>) were prepared by arylation of BCl<sub>3</sub> with corresponding dimethyldiarylstannanes Me<sub>2</sub>SnR<sub>2</sub>, providing a high-yield simple preparative route. Some specific Me<sub>2</sub>SnR<sub>2</sub> (R = 2-MeOC<sub>6</sub>H<sub>4</sub>, 2,6-(MeO)<sub>2</sub>C<sub>6</sub>H<sub>3</sub>, 2-CF<sub>3</sub>C<sub>6</sub>H<sub>4</sub>) failed to yield the desired diarylchloroboranes. The phosphines MePR<sub>12</sub> were lithiated and reacted with diarylchloroboranes or dialkylchloroboranes to give



bis(phosphino)borates [R<sub>2</sub>B(CH<sub>2</sub>PR<sub>12</sub>)<sub>2</sub>]- as their lithium salts, which then may be converted into tetraalkylammonium, thallium or ASN<sup>+</sup> (ASN<sup>+</sup> = 5-azoniaspiro[4.4]nonane) salts. Specifically, several new monoanionic bis(phosphino)borates are described (M = Li(TMEDA), ASN; in some salts also Et<sub>4</sub>N, Bu<sub>4</sub>N, Tl): [Ph<sub>2</sub>B(CH<sub>2</sub>PPh<sub>2</sub>)<sub>2</sub>]-M<sup>+</sup> (25), [(4-MeC<sub>6</sub>H<sub>4</sub>)<sub>2</sub>B(CH<sub>2</sub>PPh<sub>2</sub>)<sub>2</sub>]-M<sup>+</sup> (26), [(4-tBuC<sub>6</sub>H<sub>4</sub>)<sub>2</sub>B(CH<sub>2</sub>PPh<sub>2</sub>)<sub>2</sub>]-M<sup>+</sup> (27), [(4-MeOC<sub>6</sub>H<sub>4</sub>)<sub>2</sub>B(CH<sub>2</sub>PPh<sub>2</sub>)<sub>2</sub>]-M<sup>+</sup> (28), [(4-CF<sub>3</sub>C<sub>6</sub>H<sub>4</sub>)<sub>2</sub>B(CH<sub>2</sub>PPh<sub>2</sub>)<sub>2</sub>]-M<sup>+</sup> (29), [Cy<sub>2</sub>B(CH<sub>2</sub>PPh<sub>2</sub>)<sub>2</sub>]-M<sup>+</sup> (30), [Ph<sub>2</sub>B(CH<sub>2</sub>P{4-tBuC<sub>6</sub>H<sub>4</sub>})<sub>2</sub>]-M<sup>+</sup> (31), [(4-MeOC<sub>6</sub>H<sub>4</sub>)<sub>2</sub>B(CH<sub>2</sub>P{4-tBuC<sub>6</sub>H<sub>4</sub>})<sub>2</sub>]-M<sup>+</sup> (32), [Ph<sub>2</sub>B(CH<sub>2</sub>P{4-CF<sub>3</sub>C<sub>6</sub>H<sub>4</sub>})<sub>2</sub>]-M<sup>+</sup> (33); [Ph<sub>2</sub>B(CH<sub>2</sub>PMe<sub>2</sub>(BH<sub>3</sub>))<sub>2</sub>]-M<sup>+</sup> (34); [Ph<sub>2</sub>B(CH<sub>2</sub>P(S)Me<sub>2</sub>)<sub>2</sub>]-M<sup>+</sup> (35); [Ph<sub>2</sub>B(CH<sub>2</sub>PiPr<sub>2</sub>)<sub>2</sub>]-M<sup>+</sup> (36); [Ph<sub>2</sub>B(CH<sub>2</sub>PtBu<sub>2</sub>)<sub>2</sub>]-M<sup>+</sup> (37); [(3,5-Me<sub>2</sub>C<sub>6</sub>H<sub>3</sub>)<sub>2</sub>B(CH<sub>2</sub>PtBu<sub>2</sub>)<sub>2</sub>]-M<sup>+</sup> (38). The chelation of bis(diarylphosphino)borates derivs. 25-33 and 36 to platinum was examined by preparation of generation of a series of platinum di-Me complexes

[[R<sub>2</sub>B(CH<sub>2</sub>PR<sub>12</sub>)<sub>2</sub>]PtMe<sub>2</sub>]-M<sup>+</sup>, which were then converted to corresponding carbonyl zwitterionic complexes [R<sub>2</sub>B(CH<sub>2</sub>PR<sub>12</sub>)<sub>2</sub>PtMe(CO)]. The electronic effects of substituted bis(phosphino)borates on the carbonyl stretching frequency of neutral platinum alkyl carbonyl complexes were studied by IR spectroscopy. Crystal structures of bis(phosphino)borates 25[Li], 25[Tl], 36[Li], 37[Li] and 38[Tl] are reported.

RX(15) OF 262



NOTE: safety - high pressure

CON: STAGE(1) room temperature

STAGE(2) room temperature -> 60 deg C; 20 hours, 60 deg C

REFERENCE COUNT: 121 THERE ARE 121 CITED REFERENCES AVAILABLE FOR THIS RECORD. ALL CITATIONS AVAILABLE IN THE REFORMAT

L4 ANSWER 15 OF 50 CASREACT COPYRIGHT 2008 ACS on STN

ACCESSION NUMBER: 138:238300 CASREACT

TITLE: Preparation of anionic borate ligands and transition metal zwitterionic complexes formed therefrom as catalysts for organometallic transformations

INVENTOR(S): Peters, Jonas C.; Thomas, John C.; Lu, Connie; Betley, Theodore A.

PATENT ASSIGNEE(S): California Institute of Technology, USA

SOURCE: U.S. Pat. Appl. Publ., 19 pp.

CODEN: USXXCO

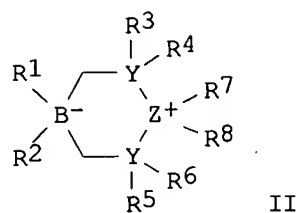
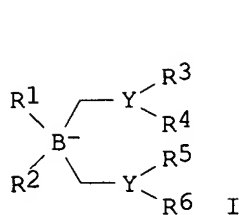
DOCUMENT TYPE: Patent

LANGUAGE: English

FAMILY ACC. NUM. COUNT: 1

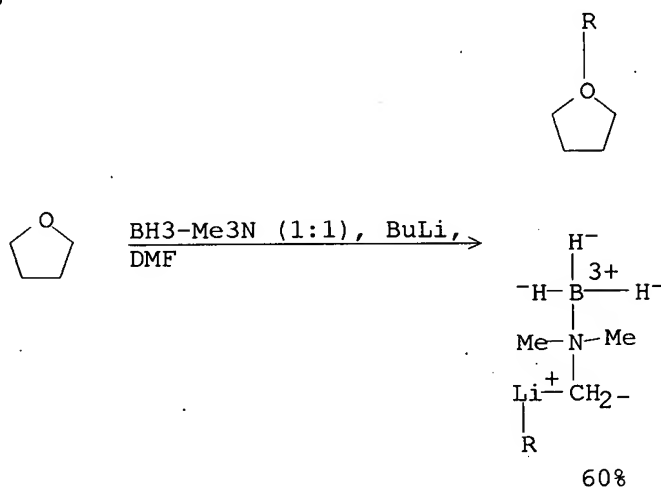
PATENT INFORMATION:

PATENT NO.	KIND	DATE	APPLICATION NO.	DATE
US 20030050493	A1	20030313	US 2002-112679	20020328
US 6649801	B2	20031118		
PRIORITY APPLN. INFO.:			US 2001-280638P	20010330
OTHER SOURCE(S):		MARPAT 138:238300		
GI				



AB Anionic borate ligands [I; wherein R1, R2, R3, R4, R5, R6, independently = aryl or alkyl; Y = P, N] and transition metal zwitterionic complexes [II; wherein R7, R8, independently = halo, alkyl, aryl, neutral donor ligands; Z = Pt, Rh, Pd, Ir, Ru, etc.] formed therefrom, were prepared. For example, complexation of I (R1 = R2 = R3 = R4 = R5 = R6 = Ph; Y = P) with (COD)Pt(Me)<sub>2</sub> afforded 98% II (R7 = R8 = Me; Z = Pt) (all as 5-azonia-spiro[4.4]nonane salts). The prepared compds. were useful as catalysts for such reactions as hydroboration, hydrosilylation and intramol. hydroacylation. For example, II (R1 = R2 = Ph; R3 = R4 = R5 = R6 = Me; R7 = R8 = NCCH<sub>3</sub>; Y = N; Z = Rh) catalyzed the hydroboration of styrene with catecholborane in 93% yield.

RX(1) OF 18



CON: STAGE(1) 5 minutes, room temperature; 5 hours, room temperature

L4 ANSWER 16 OF 50 CASREACT COPYRIGHT 2008 ACS on STN

ACCESSION NUMBER: 138:39323 CASREACT

TITLE: Synthesis and characterization of lanthanide complexes involving silylene-bridged fluorenyl

AUTHOR(S): Luo, Mei; Ma, Huai-Zhu; Su, Qing-De; Hu, Nai-Liang; Du, Bao-Jun

CORPORATE SOURCE: Dep. of Chem., Univ. of Sci. and Technol. of China, Hefei, Anhui, 230 026, Peop. Rep. China

SOURCE: Asian Journal of Chemistry (2002), 14(3-4), 1469-1473  
CODEN: AJCHEW; ISSN: 0970-7077

PUBLISHER: Asian Journal of Chemistry

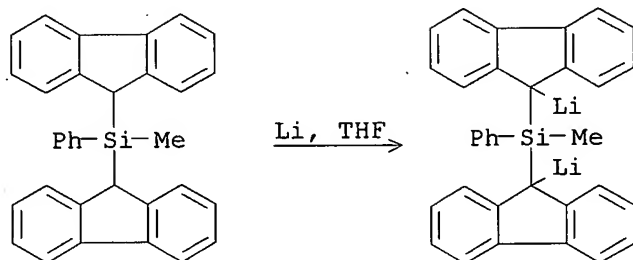
DOCUMENT TYPE: Journal

LANGUAGE: English

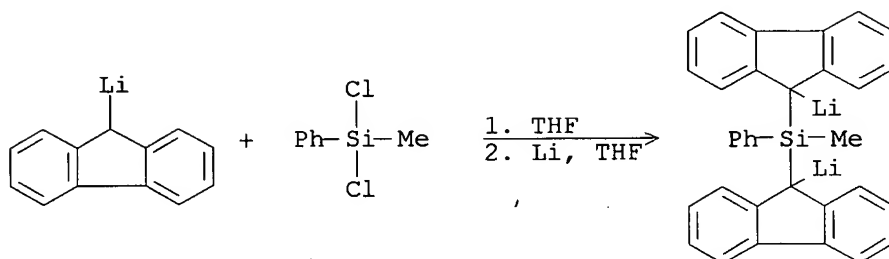
AB By the reaction of bis(9-lithiofluorenyl)(methyl)(phenyl)silane with LnCl<sub>3</sub> (Ln = Yb, Dy, Pr, La, Sm, Nd), six new lanthanide complexes were

synthesized. All the complexes were characterized by elemental anal., IR spectra and mass spectra.

RX(8) OF 21



RX(9) OF 21 - 2 STEPS



REFERENCE COUNT: 9 THERE ARE 9 CITED REFERENCES AVAILABLE FOR THIS RECORD. ALL CITATIONS AVAILABLE IN THE RE FORMAT

L4 ANSWER 17 OF 50 CASREACT COPYRIGHT 2008 ACS on STN

ACCESSION NUMBER: 136:355263 CASREACT

TITLE: Sodium-Potassium Alloy for the Reduction of Monoalkyl Aluminum(III) Compounds

AUTHOR(S): Schormann, Mark; Klimek, Klaus S.; Hatop, Hagen; Varkey, Saji P.; Roesky, Herbert W.; Lehmann, Christopher; Roepken, Cord; Herbst-Irmer, Regine; Noltemeyer, Mathias

CORPORATE SOURCE: Institut fuer Anorganische Chemie, Georg August Universitaet Goettingen, Goettingen, 37077, Germany

SOURCE: Journal of Solid State Chemistry (2001), 162(2), 225-236

CODEN: JSSCBI; ISSN: 0022-4596

PUBLISHER: Academic Press

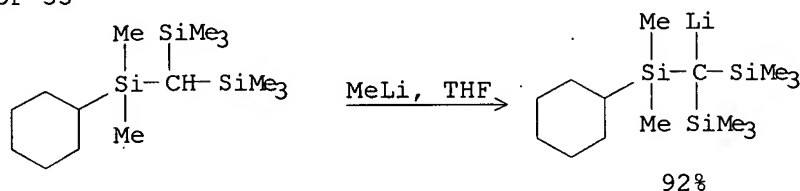
DOCUMENT TYPE: Journal

LANGUAGE: English

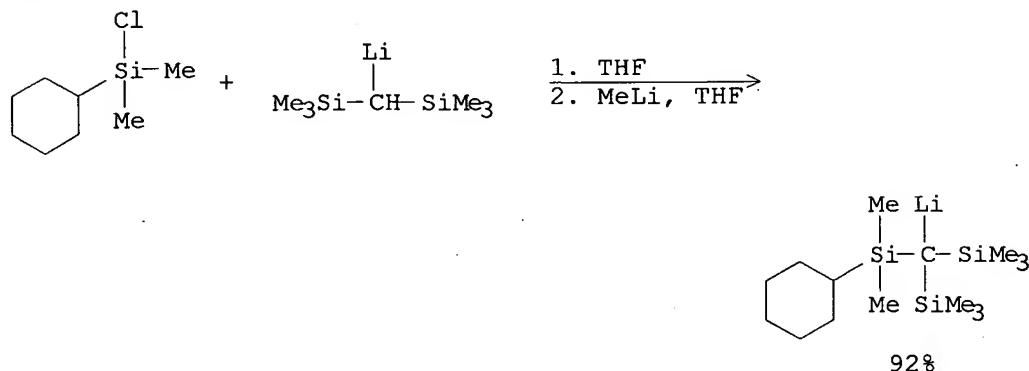
AB Monoalkylaluminum(III) compds. of the type  $RAlX_2$  ( $R = Cp^* (C_5Me_5)$ ,  $X = Cl, Br, I$  (1-3);  $(BisAlCl_2)_2$  ( $Bis = (Me_3Si)_2CH$ ) (5);  $TrisSi [(Me_3Si)_3Si]$ ,  $X = Cl, Br, I$  (6-8);  $CycTris [(CycMe_2Si)(Me_3Si)_2C]$ ,  $X = Me, F, Cl, Br, I$  (11-15)) were prepared and characterized by NMR-, IR-, and mass spectroscopy as well as elemental anal. The single-crystal x-ray structures of  $Cp^*AlBr_2$ ,  $TrisSiAlX_2 \cdot THF$  ( $X = Cl, Br, I$ ),  $CycTrisAlX_2 \cdot THF$  ( $X = Me, Cl, Br, I$ ), and  $[CycTrisAl(\mu-O(CH_2)_3CH_2)]_2$  are reported. The monoalkylaluminum(I) compound  $(Cp^*Al)_4$  (4) was isolated after the reduction of  $Cp^*AlX_2$  ( $X = Cl, Br, I$ ) using a Na/K alloy. The yield of  $(Cp^*Al)_4$  has been improved compared to the previously reported method. However, completely different products were obtained when the THF adducts of  $TrisSiAlI_2 \cdot THF$  and  $CycTrisAlI_2 \cdot THF$  were used for the reduction with Na/K alloy. In the former case, the cleavage

of the Si-Al bond was observed with the formation of elemental aluminum and (TrisSi)2, while in the latter the THF ring opening reaction occurred. CycTrisAlF2·THF was prepared by reacting CycTrisAlMe2·THF with Me3SnF. (c) 2001 Academic Press.

RX(9) OF 35



RX(17) OF 35 - 2 STEPS



REFERENCE COUNT: 60 THERE ARE 60 CITED REFERENCES AVAILABLE FOR THIS RECORD. ALL CITATIONS AVAILABLE IN THE RE FORMAT

L4 ANSWER 18 OF 50 CASREACT COPYRIGHT 2008 ACS on STN

ACCESSION NUMBER: 136:263225 CASREACT

TITLE: Synthesis of metallocenes of zirconium, hafnium, manganese, iron, tin, lead and half-sandwich complexes of rhodium and iridium containing the ligands ( $\eta$ -C5R4CR'2PMe2), where R and R' may be H or Me

AUTHOR(S): Bellabarba, Ronan M.; Clancy, Gerald P.; Gomes, Pedro T.; Martins, Ana M.; Rees, Leigh H.; Green, Malcolm L. H.

CORPORATE SOURCE: Wolfson Catalysis Centre, Inorganic Chemistry Laboratory, Oxford, OX1 3QR, UK

SOURCE: Journal of Organometallic Chemistry (2001), 640(1-2), 93-112

CODEN: JORCAI; ISSN: 0022-328X

PUBLISHER: Elsevier Science S.A.

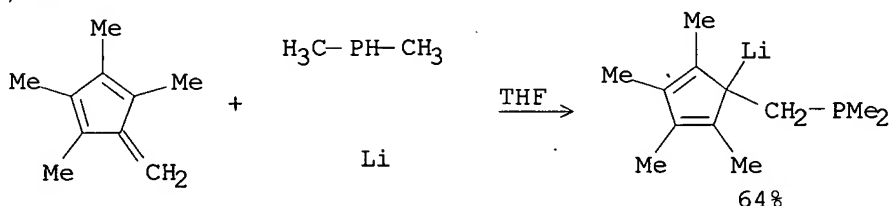
DOCUMENT TYPE: Journal

LANGUAGE: English

AB The dimethylphosphino substituted cyclopentadienyl precursor compds. [M(C5Me4CH2PMe2)], where M = Li (1), Na (2), or K (3), and [Li(C5H4CR'2PMe2)], where R'2 = Me2 (4), or (CH2)5 (5), [HC5Me4CH2PMe2H]X, where X = Cl (6) or PF6 (7) and [HC5Me4CH2PMe2] (8), are described. They were used to prepare new metallocene compds., of which representative examples are [Fe( $\eta$ -C5R4CR'2PMe2)2], where R = Me, R' = H (9); R = H and R'2 = Me2 (10), or (CH2)5 (11), [Fe( $\eta$ -C5H4CMe2PMe3)2]I2 (12), [Fe( $\eta$ -C5Me4CH2P(O)Me2)2] (13), [Zr( $\eta$ -C5R4CR'2PMe2)2Cl2], where R = H, R' = Me (14), or R = Me, R' = H (15), [Hf( $\eta$ -C5H4CMe2PMe2)2Cl2] (16), [Zr( $\eta$ -C5H4CMe2PMe2)2Me2] (17), {[Zr( $\eta$ -

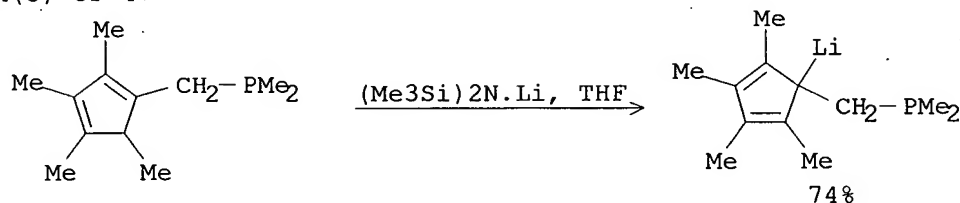
C5Me4CH2PMe2)2]Cl]((C6F5)3BClB(C6F5)3) (18), [Zr{(η-C5Me4CH2PMe2)2Cl2}PtI2] (19), [Mn(η-C5Me4CH2PMe2)2] (20), [Mn{(η-C5Me4CH2PMe2)B(C6F5)3}2] (21), [Pb(η-C5H4CMe2PMe2)2] (23), [Sn(η-C5H4CMe2PMe2)2] (24), [Pb(η-C5H4CMe2PMe2B(C6F5)3)2] (25), [Pb(η-C5H4CMe2PMe2)2PtI2] (26), [Rh(η-C5Me4CH2PMe2)(C2H4)] (29), [M(η,κP-C5Me4CH2PMe2)I2], where M = Rh (30), or Ir, (31).

RX(1) OF 47



NOTE: -78.degree.

RX(3) OF 47



NOTE: -78.degree. to room temp., overnight

REFERENCE COUNT: 75 THERE ARE 75 CITED REFERENCES AVAILABLE FOR THIS RECORD. ALL CITATIONS AVAILABLE IN THE RE FORMAT

L4 ANSWER 19 OF 50 CASREACT COPYRIGHT 2008 ACS on STN

ACCESSION NUMBER: 135:92684 CASREACT

TITLE: Reaction of m-Terphenyldichlorophosphanes with Sodium Azide: Synthesis and Characterization of Stable Azidocyclophosphazenes

AUTHOR(S): Wehmschulte, Rudolf J.; Khan, Masood A.; Hossain, Shawn I.

CORPORATE SOURCE: Department of Chemistry and Biochemistry, University of Oklahoma, Norman, OK, 73019, USA

SOURCE: Inorganic Chemistry (2001), 40(12), 2756-2762  
CODEN: INOCAJ; ISSN: 0020-1669

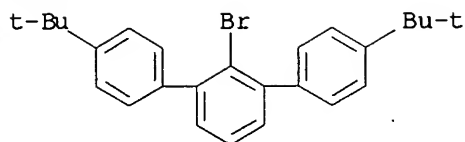
PUBLISHER: American Chemical Society

DOCUMENT TYPE: Journal

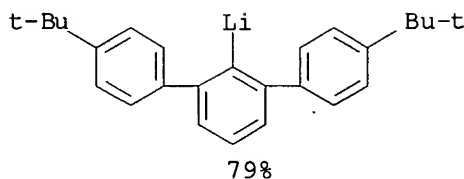
LANGUAGE: English

AB Reaction of the m-terphenyldichlorophosphanes 2,6-(2-MeC6H4)2C6H3PCl2 (1), 2,6-(4-t-BuC6H4)2C6H3PCl2 (2), or 2,6-Mes2C6H3PCl2 (3) with excess NaN3 in acetonitrile at room temperature afforded the corresponding bisazidophosphanes 2,6-(2-MeC6H4)2C6H3P(N3)2, 2,6-(4-t-BuC6H4)2C6H3P(N3)2 (5), or 2,6-Mes2C6H3P(N3)2 (6) (Mes = 2,4,6-Me3C6H2), resp. These compds. are thermally labile and decompose into a number of azidophosphazenes. The azidocyclophosphazenes [NP(N3)(C6H3(4-t-BuC6H4)2-2,6)]3 (4) and [NP(N3)C6H3Mes2-2,6]2 (8) were isolated from these mixts. All compds. were characterized by 1H, 13C, 31P NMR and IR spectroscopy. Crystal structures of 2, 4, and 8 were determined

RX(6) OF 11



BuLi, Hexane →



REFERENCE COUNT: 37 THERE ARE 37 CITED REFERENCES AVAILABLE FOR THIS RECORD. ALL CITATIONS AVAILABLE IN THE RE FORMAT

L4 ANSWER 20 OF 50 CASREACT COPYRIGHT 2008 ACS on STN

ACCESSION NUMBER: 134:86345 CASREACT

TITLE: Synthesis, Characterization, and Polymerization Properties of Bis(2-menthylindenyl)zirconium Dichloride and Bis(2-menthyl-4,7-dimethylindenyl)zirconium Dichloride

AUTHOR(S): Halterman, Ronald L.; Fahey, Darryl R.; Bailly, Eric F.; Dockter, David W.; Stenzel, Oleg; Shipman, Jason L.; Khan, Masood A.; Dechert, Sebastian; Schumann, Herbert

CORPORATE SOURCE: Department of Chemistry and Biochemistry, University of Oklahoma, Norman, OK, 73019, USA

SOURCE: Organometallics (2000), 19(25), 5464-5470  
CODEN: ORGND7; ISSN: 0276-7333

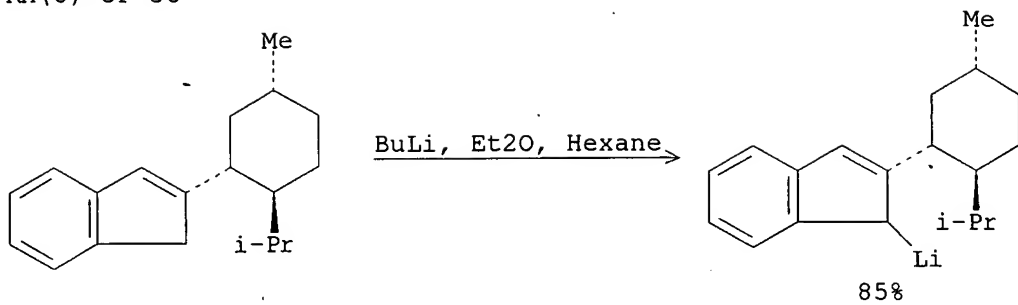
PUBLISHER: American Chemical Society

DOCUMENT TYPE: Journal

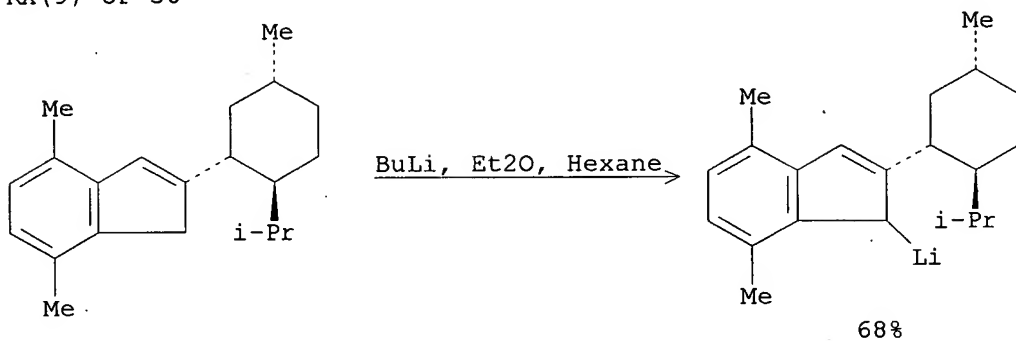
LANGUAGE: English

AB The tetrakis(triphenylphosphine)palladium-catalyzed cross coupling of 2-bromoindene or 2-bromo-4,7-dimethylindene with menthylmagnesium chloride gave the novel ligands 2-menthylindene (10) and 2-menthyl-4,7-dimethylindene (11) in 66 and 57% yields. These indenenes were deprotonated with n-BuLi to give isolated indenyllithium complexes which were metalated with zirconium tetrachloride to give bis(2-menthylindenyl)zirconium dichloride (14) and bis(2-menthyl-4,7-dimethylindenyl)zirconium dichloride (15) in 40 and 66% yields. Both complexes formed as their single possible stereoisomer and were characterized by x-ray crystallog. and MS, NMR, and IR spectroscopy. The variable-temperature <sup>1</sup>H NMR spectrum of bis(2-menthyl-4,7-dimethylindenyl)zirconium dichloride (15) showed that it interconverts with a rotational activation barrier of about 12.5 ± 0.5 kcal/mol, and that a 60:40 mixture of C<sub>2</sub>-sym. diastereomeric conformations was present at -50°. The <sup>1</sup>H NMR spectrum of bis(2-menthylindenyl)zirconium dichloride (14) was invariant between -50 and +50°. Complexes 14 and 15 polymerized propene in the presence of methylaluminumoxane (MAO) and hydrogen (10 960 and 33 750 g PP/g Zr h, resp.). The mol. wts. and stereoregularity of the polymers produced were low.

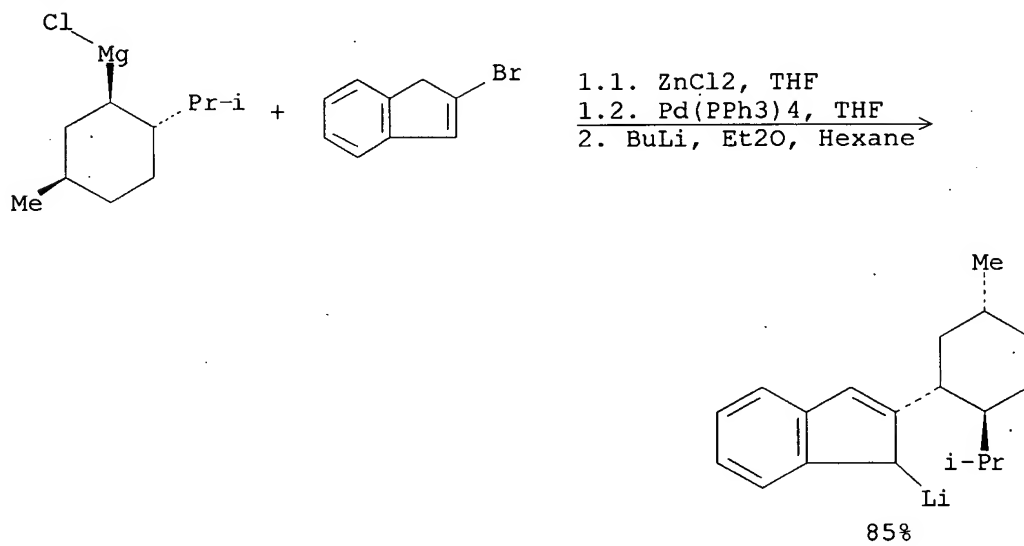
RX(8) OF 36



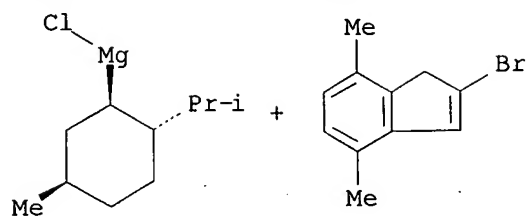
RX(9) OF 36



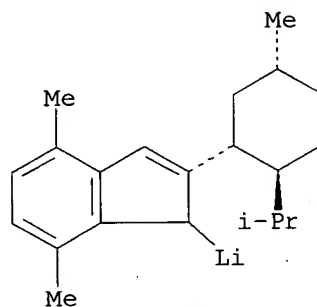
RX(17) OF 36 - 2 STEPS



RX(18) OF 36 - 2 STEPS

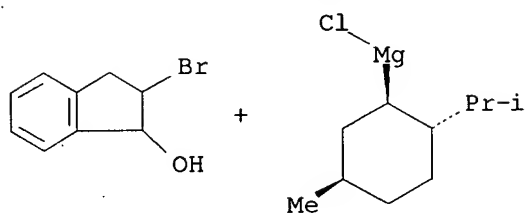


1.1.  $\text{ZnCl}_2$ , THF  
 1.2.  $\text{Pd}(\text{PPh}_3)_4$ , THF  
 2. BuLi, Et<sub>2</sub>O, Hexane

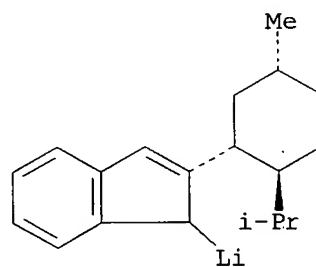


68%

RX(25) OF 36 - 3 STEPS



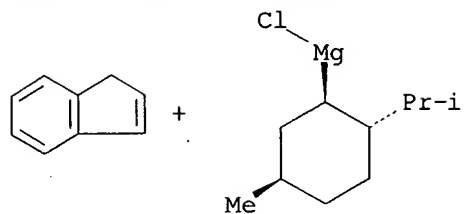
1. TsOH, PhMe  
 2.1.  $\text{ZnCl}_2$ , THF  
 2.2.  $\text{Pd}(\text{PPh}_3)_4$ , THF  
 3. BuLi, Et<sub>2</sub>O, Hexane



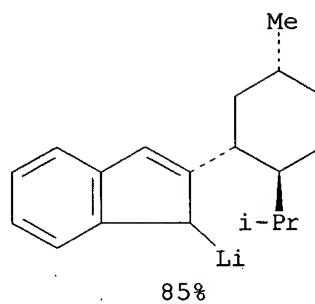
85%



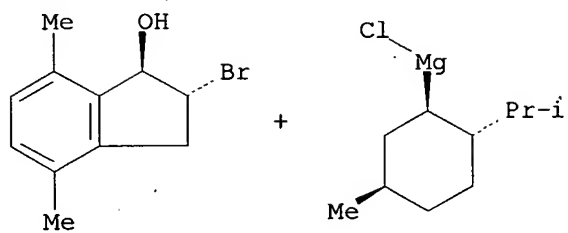
RX(26) OF 36 - 4 STEPS



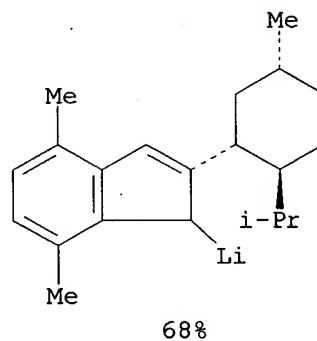
1. Water,  
Bromosuccinimide,  
DMSO
2. TsOH, PhMe
- 3.1. ZnCl<sub>2</sub>, THF
- 3.2. Pd(PPh<sub>3</sub>)<sub>4</sub>, THF
4. BuLi, Et<sub>2</sub>O, Hexane



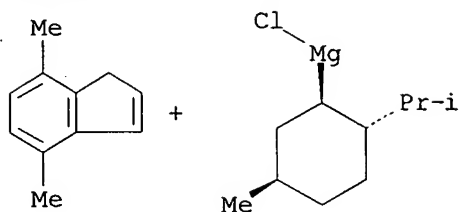
RX(27) OF 36 - 3 STEPS



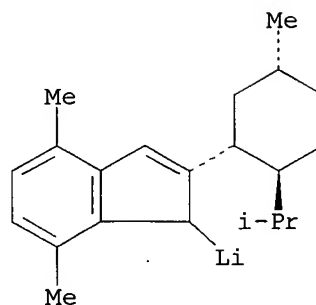
1. TsOH, PhMe
- 2.1. ZnCl<sub>2</sub>, THF
- 2.2. Pd(PPh<sub>3</sub>)<sub>4</sub>, THF
3. BuLi, Et<sub>2</sub>O, Hexane



RX(28) OF 36 - 4 STEPS

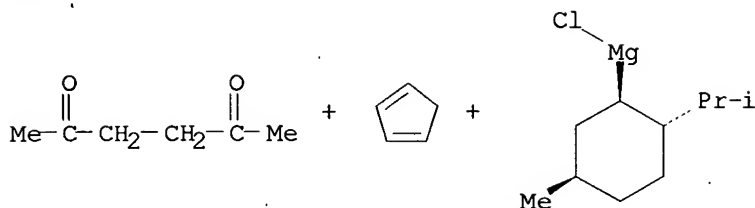


1. Water,  
Bromosuccinimide,  
DMSO
2. TsOH, PhMe
- 3.1. ZnCl<sub>2</sub>, THF
- 3.2. Pd(PPh<sub>3</sub>)<sub>4</sub>, THF
4. BuLi, Et<sub>2</sub>O, Hexane

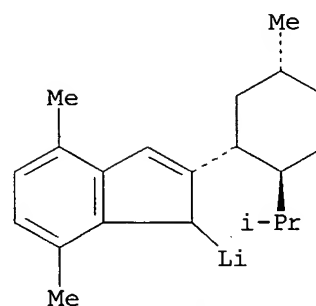


68%

RX(33) OF 36 - 5 STEPS



- 1.1. NaOMe, MeOH
2. Water,  
Bromosuccinimide,  
DMSO
3. TsOH, PhMe
- 4.1. ZnCl<sub>2</sub>, THF
- 4.2. Pd(PPh<sub>3</sub>)<sub>4</sub>, THF
5. BuLi, Et<sub>2</sub>O, Hexane



68%

REFERENCE COUNT:

45

THERE ARE 45 CITED REFERENCES AVAILABLE FOR THIS RECORD. ALL CITATIONS AVAILABLE IN THE RE FORMAT

L4 ANSWER 21 OF 50 CASREACT COPYRIGHT 2008 ACS on STN

ACCESSION NUMBER: 132:293847 CASREACT

TITLE: Synthesis and properties of metallocene and half-sandwich complexes with pyridine-containing bridges or side chains

AUTHOR(S): Schliessburga, Christine; Thiele, Karl-Heinz; Lindner, Birgit; Bruser, Wolfgang

CORPORATE SOURCE: Institut für Anorganische Chemie, Martin-Luther-

SOURCE:

Universitat, Halle, D-06120, Germany  
Zeitschrift fuer Anorganische und Allgemeine Chemie  
(2000), 626(3), 741-746  
CODEN: ZAACAB; ISSN: 0044-2313

PUBLISHER:

Wiley-VCH Verlag GmbH

DOCUMENT TYPE:

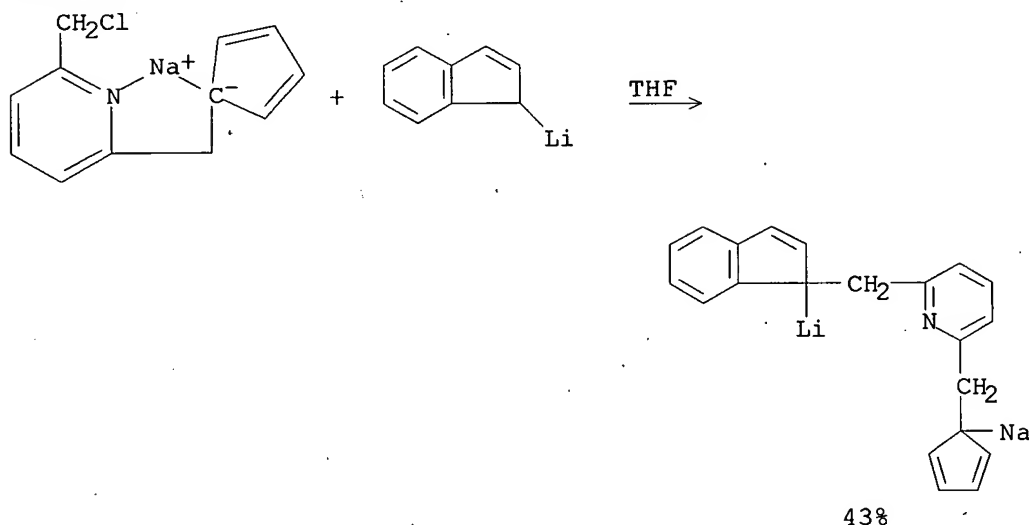
Journal

LANGUAGE:

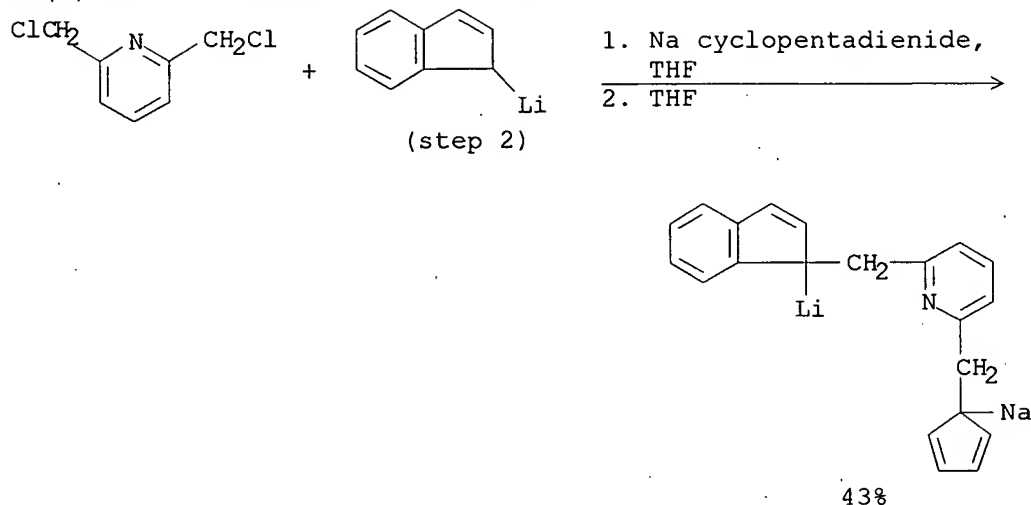
German

AB 2,6-Bis(chloromethyl)pyridine (1) reacts with 4 equiv indenyllithium with formation of dilithium 2,6-bis(methyleneindenyl)pyridine, from which the corresponding metallocene dichlorides can be obtained upon treatment with  $MCl_4 \cdot 2THF$  ( $M = Zr, Hf$ ). Upon reaction of 1 with 2 equiv  $C_5H_5Na$ , only one Cl atom is replaced by a  $C_5H_5Na$  unit. Subsequent reactions with indenyllithium and  $ZrCl_4 \cdot 2THF$  give unsym. [2-(cyclopentadienylmethyl)-6-(inden-1-ylmethyl)pyridine]zirconium dichloride. Picolinylcyclopentadiene and 1-picolinylindene were synthesized from 2-(chloromethyl)pyridine.HCl and  $C_5H_5Na$  or indenyllithium, resp., and are transformed into the corresponding half-sandwich complexes  $(C_5H_4N-CH_2C_5H_4)MCl_3$  ( $M = Ti, Zr$ ) and  $(C_5H_4-CH_2C_9H_6)ZrCl_3$ . The compds. were characterized by elemental anal.,  $^1H$  NMR, IR, Raman, and mass spectra. N→M interactions are discussed.

RX(2) OF 7



RX(5) OF 7 - 2 STEPS



REFERENCE COUNT: 25 THERE ARE 25 CITED REFERENCES AVAILABLE FOR THIS RECORD. ALL CITATIONS AVAILABLE IN THE RE FORMAT

L4 ANSWER 22 OF 50 CASREACT COPYRIGHT 2008 ACS on STN

ACCESSION NUMBER: 131:271922 CASREACT

TITLE: P-H-functionalised phosphinocyclopentadienes:  
1-SiMe<sub>2</sub>PHCy-2,3,4,5-Me<sub>4</sub>C<sub>5</sub>H, Li<sub>2</sub>[(C<sub>5</sub>H<sub>4</sub>)SiMe<sub>2</sub>PCy],  
Li[(C<sub>5</sub>H<sub>4</sub>)CMe<sub>2</sub>PHPh] and Li<sub>2</sub>[(C<sub>5</sub>H<sub>4</sub>)CH<sub>2</sub>CH<sub>2</sub>PPh]

AUTHOR(S): Koch, Thomas; Hey-Hawkins, Evamarie

CORPORATE SOURCE: Institut für Anorganische Chemie der Universität  
Leipzig, Leipzig, D-04103, Germany

SOURCE: Polyhedron (1999), 18(16), 2113-2116  
CODEN: PLYHDE; ISSN: 0277-5387

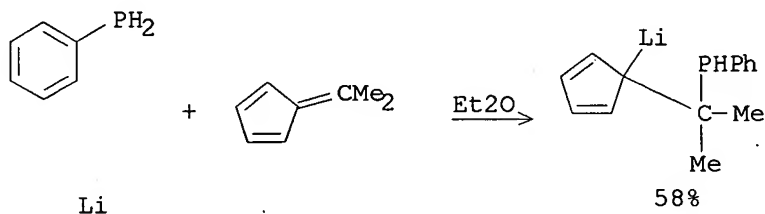
PUBLISHER: Elsevier Science Ltd.

DOCUMENT TYPE: Journal

LANGUAGE: English

AB Several P-H-functionalized phosphinocyclopentadienes and Li salts thereof were prepared: 1-SiMe<sub>2</sub>PHCy-2,3,4,5-Me<sub>4</sub>C<sub>5</sub>H (1) (Cy = C<sub>6</sub>H<sub>11</sub>), Li<sub>2</sub>[(C<sub>5</sub>H<sub>4</sub>)SiMe<sub>2</sub>PCy] (1a), Li[(C<sub>5</sub>H<sub>4</sub>)CMe<sub>2</sub>PHPh] (2) and Li<sub>2</sub>[(C<sub>5</sub>H<sub>4</sub>)CH<sub>2</sub>CH<sub>2</sub>PPh] (3). 1 Was obtained from 1-SiMe<sub>2</sub>Cl-2,3,4,5-Me<sub>4</sub>C<sub>5</sub>H and LiPHCy. Treatment of 1 with MeLi yields the corresponding dilithio salt 1a. 6,6-Dimethylfulvene reacts with LiPHPh to give 2. The three-membered ring of spiro[2,4]hepta-4,6-diene is cleaved by LiPHPh. Successive treatment of the intermediate monolithio salt with MeLi gives 3. Compds. 1-3 were characterized spectroscopically (1H, 31P, 13C, 7Li NMR; IR).

RX(1) OF 3



REFERENCE COUNT: 43 THERE ARE 43 CITED REFERENCES AVAILABLE FOR THIS RECORD. ALL CITATIONS AVAILABLE IN THE RE FORMAT

L4 ANSWER 23 OF 50 CASREACT COPYRIGHT 2008 ACS on STN

ACCESSION NUMBER: 123:169805 CASREACT

TITLE: Bis(alkynyl) and alkynyl-vinylidene iron(II) complexes with monodentate phosphite ligands

AUTHOR(S): Albertin, Gabriele; Antoniutti, Stefano; Bordignon, Emilio; Del Ministro, Elena; Ianelli, Sandra; Pelizzi, Giancarlo

CORPORATE SOURCE: Dip. Chimica, Univ. Venezia, Venice, 30123, Italy

SOURCE: Journal of the Chemical Society, Dalton Transactions: Inorganic Chemistry (1995), (11), 1783-9  
CODEN: JCDTBI; ISSN: 0300-9246

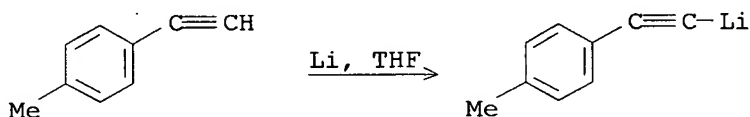
PUBLISHER: Royal Society of Chemistry

DOCUMENT TYPE: Journal

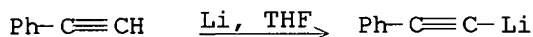
LANGUAGE: English

AB The bis(alkynyl) derivs.  $[\text{Fe}(\text{C.tplbond.CR})_2\text{L}_4]$   $[\text{R} = \text{Ph}, \text{p-tolyl}, \text{CMe}_3; \text{L} = \text{P}(\text{OMe})_3, \text{P}(\text{OEt})_3, \text{PPh}(\text{OEt})_2]$  were prepared and their protonation and methylation reactions with  $\text{HBF}_4$  and  $\text{CF}_3\text{SO}_3\text{Me}$  afforded alkynyl-vinylidene cations  $[\text{Fe}(\text{C.tplbond.CR})\{\text{:C:C(H)R}\}\text{L}_4]^+$  and  $[\text{Fe}(\text{C.tplbond.CR})\{\text{:C:C(Me)R}\}\text{L}_4]^+$ , resp. The aryldiazovinylidene  $[\text{Fe}(\text{C.tplbond.CR})\{\text{:C:C(N:NC}_6\text{H}_4\text{Me-p)Ph}\}\{\text{P}(\text{OEt})_3\}_4]\text{BPh}_4$  was also prepared. The complexes were characterized by IR and  $^1\text{H}$ ,  $^{31}\text{P}$  and  $^{13}\text{C}$  NMR spectra and the crystal structure of  $[\text{Fe}(\text{C.tplbond.CPh})\{\text{:C:C(H)Ph}\}\{\text{P}(\text{OEt})_3\}_4]\text{BF}_4$  has been determined. The reactivity of the new vinylidene complexes was studied and showed the rearrangement in solution of the  $[\text{Fe}(\text{C.tplbond.CR})\{\text{:C:C(H)R}\}\text{L}_4]^+$  cations to enynyl  $[\text{Fe}(\eta^3\text{-RC}_3\text{CHR})\text{L}_4]^+$  derivs. only in the case of  $\text{L} = \text{PPh}(\text{OEt})_2$ . Deprotonation with base giving  $[\text{Fe}(\text{C.tplbond.CR})_2\text{L}_4]$  as well as substitution of the vinylidene ligand in  $[\text{Fe}(\text{C.tplbond.CR})\{\text{:C:C(H)R}\}\text{L}_4]^+$  cations by CO and  $\text{CNC}_6\text{H}_4\text{Me-p}$  giving  $[\text{Fe}(\text{C.tplbond.CR})(\text{CO})\{\text{P}(\text{OEt})_3\}_4]^+$  and  $[\text{Fe}(\text{C.tplbond.CR})(\text{p-MeC}_6\text{H}_4\text{NC})\{\text{P}(\text{OEt})_3\}_4]^+$  derivs. are also discussed.

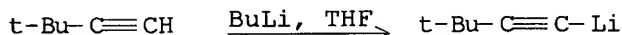
RX(20) OF 62



RX(23) OF 62



RX(24) OF 62



L4 ANSWER 24 OF 50 CASREACT COPYRIGHT 2008 ACS on STN

ACCESSION NUMBER: 120:134001 CASREACT

TITLE: Fulvalenes. 63. Synthesis and pyrolysis of a triafulvene precursor

AUTHOR(S): Muehlebach, Michel; Neuenschwander, Markus; Engel, Peter

CORPORATE SOURCE: Inst. Org. Chem., Univ. Bern, Bern, CH-3012, Switz.

SOURCE: Helvetica Chimica Acta (1993), 76(5), 2089-110  
CODEN: HCACAV; ISSN: 0018-019X

DOCUMENT TYPE: Journal

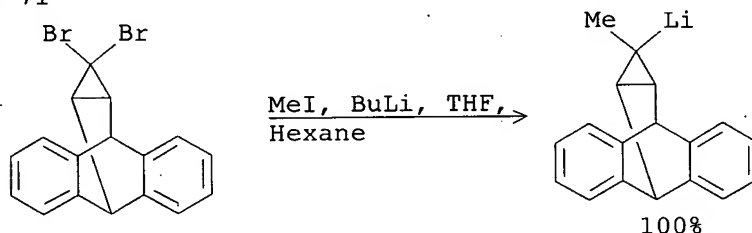
LANGUAGE: German

GI

\* STRUCTURE DIAGRAM TOO LARGE FOR DISPLAY - AVAILABLE VIA OFFLINE PRINT \*

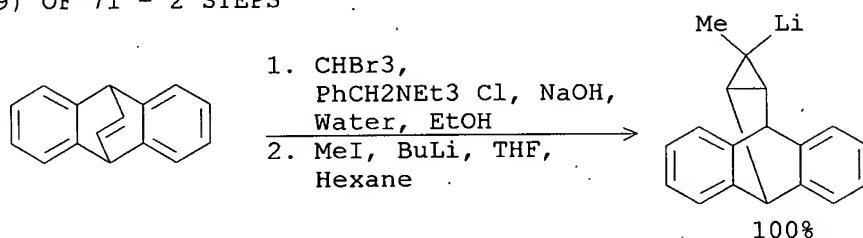
AB In view of retro-Diels-Alder reactions (RDA reactions), the triafulvene precursor I has been prepared by dibromocyclopropane addition to dibenzobarrelene (II; 44%) to give III, which was lithiated, methylated and eliminated HBr. Reactivity of the novel bridged 1,1-dibromocyclopropane III has been explored, including redns., allylic rearrangements, and carbene dimerizations. Thermolysis of I shows that RDA reaction, although occurring in most cases, is not the predominant pathway. When I is heated in a sealed tube without solvent, two dimers IV and an isomeric C<sub>36</sub>H<sub>28</sub> compound are isolated in a total yield of 55%. Gas-phase pyrolysis of I at 400° gave 56% rearranged V.

RX(7) OF 71



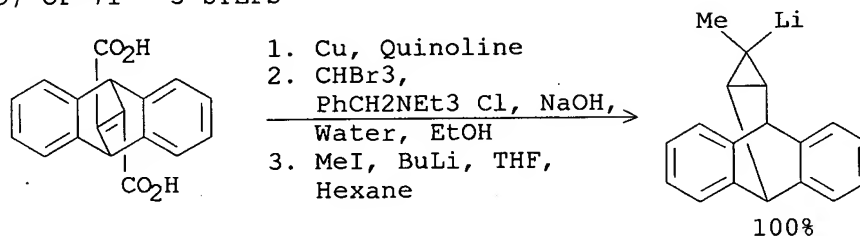
NOTE: stereoselective

RX(19) OF 71 - 2 STEPS



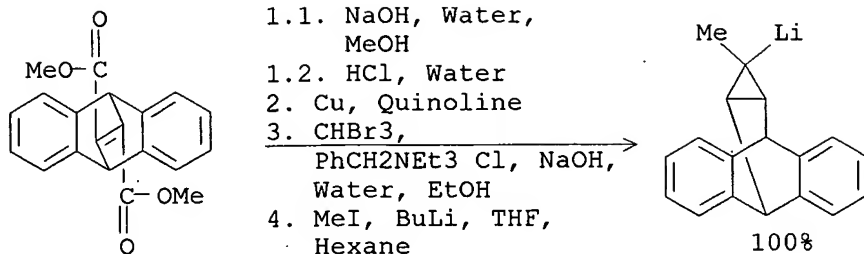
NOTE: 2) stereoselective

RX(29) OF 71 - 3 STEPS



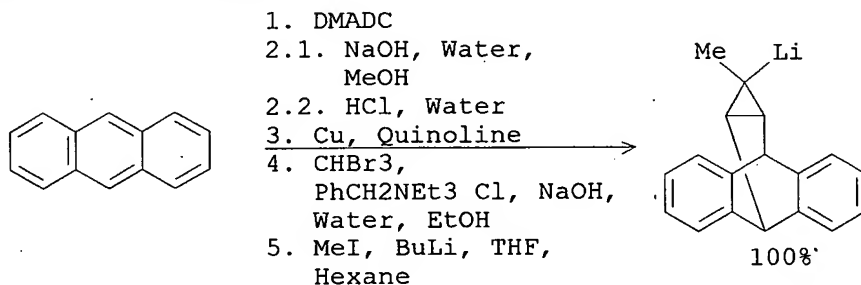
NOTE: 1) purification about the maleic anhydride adduct, 3) stereoselective

RX(33) OF 71 - 4 STEPS



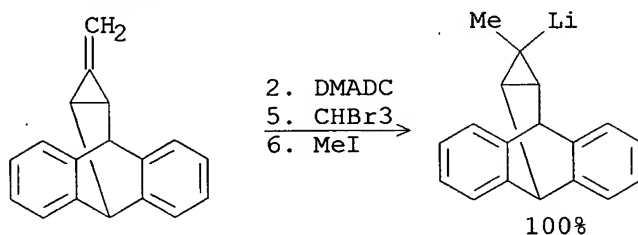
NOTE: 2) purification about the maleic anhydride adduct, 4) stereoselective

RX(48) OF 71 - 5 STEPS



NOTE: 1) thermal, 3) purification about the maleic anhydride adduct, 5) stereoselective

RX(53) OF 71 - 6 STEPS



NOTE: 1) stereoselective, thermal, low pressure, other product also detected, 2) thermal, 4) purification about the maleic anhydride adduct, 6) stereoselective

L4 ANSWER 25 OF 50 CASREACT COPYRIGHT 2008 ACS on STN

ACCESSION NUMBER:

120:107285 CASREACT

TITLE:

Bis(cyclopentadienyl)methane-bridged dinuclear complexes. 7. From mononuclear (C<sub>5</sub>H<sub>5</sub>CH<sub>2</sub>C<sub>5</sub>H<sub>4</sub>)M to unsymmetrical dinuclear M(C<sub>5</sub>H<sub>4</sub>CH<sub>2</sub>C<sub>5</sub>H<sub>4</sub>)M and heterodinuclear M(C<sub>5</sub>H<sub>4</sub>CH<sub>2</sub>C<sub>5</sub>H<sub>4</sub>)M' transition-metal complexes

AUTHOR(S):

Schneider, Dirk; Werner, Helmut

CORPORATE SOURCE:

Inst. Anorg. Chem., Univ. Wuerzburg, Wuerzburg, D-97074, Germany

SOURCE:

Organometallics (1993), 12(11), 4420-30

CODEN: ORGND7; ISSN: 0276-7333

DOCUMENT TYPE:

Journal

LANGUAGE:

English

\* STRUCTURE DIAGRAM TOO LARGE FOR DISPLAY - AVAILABLE VIA OFFLINE PRINT \*

AB The reaction of  $[\text{CH}_2(\text{C}_5\text{H}_4)_2]\text{Na}_2$ , generated in situ from  $\text{CH}_2(\text{C}_5\text{H}_5)_2$  and  $\text{NaNH}_2$  in THF at  $-70^\circ\text{C}$ , with  $[\text{RhH}(\text{C.tplbond.CR})\text{Cl}(\text{py})(\text{PiPr}_3)_2]$  ( $\text{R} = \text{Me}, \text{Ph}$ ) leads to a mixture of the mono- and dinuclear complexes  $[(\text{C}_5\text{H}_5\text{CH}_2\text{C}_5\text{H}_4)\text{Rh}(\text{:C:CHR})(\text{PiPr}_3)]$  (I) and  $\{[\text{CH}_2(\text{C}_5\text{H}_4)_2][\text{Rh}(\text{:C:CHR})(\text{PiPr}_3)]_2\}$  (II) which are separated by column chromatog. Traces of the square-planar compds.  $\text{trans-}[\text{RhCl}(\text{:C:CHR})(\text{PiPr}_3)_2]$  are also obtained. Electrophilic addition of sulfur, tosyl azide, and  $\text{CF}_3\text{CO}_2\text{H}$  to the  $\text{Rh:C}$  bond of the vinylidene complexes I and II affords thioketene-, ketenimine-, and vinyl-rhodium derivs., e.g. III. The mononuclear compds.  $[(\text{C}_5\text{H}_5\text{CH}_2\text{C}_5\text{H}_4)\text{MLL}']$ , e.g. IV, are prepared from  $(\text{C}_5\text{H}_5\text{CH}_2\text{C}_5\text{H}_4)\text{Na}$  and the corresponding rhodium(I) and iridium(I) precursors; in these reactions small amts. of dinuclear  $[\text{CH}_2(\text{C}_5\text{H}_4)_2]\text{M}_2$  complexes ( $\text{M} = \text{Rh}, \text{Ir}$ ) are also obtained. The synthesis of the mixed-metal compds., e.g. V, has been achieved from either the cyclopentadiene derivs. or the lithiated compds.  $[(\text{LiC}_5\text{H}_4\text{CH}_2\text{C}_5\text{H}_4)\text{MLL}']$ . Related unsym. dirhodium complexes  $\{[\text{CH}_2(\text{C}_5\text{H}_4)_2][\text{RhLL}'][\text{Rh}(\text{PhC.tplbond.CPh})(\text{PiPr}_3)]\}$  are prepared by a similar route.

RX(9) OF 21 - REACTION DIAGRAM NOT AVAILABLE

RX(19) OF 21 - REACTION DIAGRAM NOT AVAILABLE

L4 ANSWER 26 OF 50 CASREACT COPYRIGHT 2008 ACS on STN

ACCESSION NUMBER: 117:8150 CASREACT

TITLE: Bis(cyclopentadienyl)methane-bridged binuclear complexes. V. Heteronuclear cobalt/rhodium, cobalt/iridium, rhodium/iridium, and titanium/iridium complexes with the bis(cyclopentadienyl)methane dianion as the bridging ligand

AUTHOR(S): Werner, Helmut; Schneider, Dirk; Schulz, Michael

CORPORATE SOURCE: Inst. Anorg. Chem., Univ. Wuerzburg, Wuerzburg, W-8700, Germany

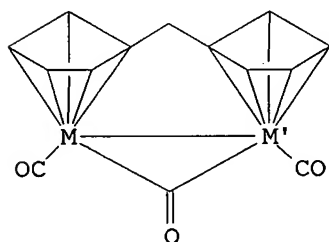
SOURCE: Chemische Berichte (1992), 125(5), 1017-22

CODEN: CHBEAM; ISSN: 0009-2940

DOCUMENT TYPE: Journal

LANGUAGE: German

GI



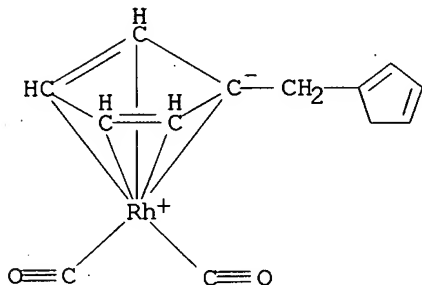
IV

AB The lithium and sodium salts of the  $[\text{C}_5\text{H}_5\text{CH}_2\text{C}_5\text{H}_4]^-$  anion react with  $[\text{Co}(\text{CO})_4\text{I}]$ ,  $[\text{Rh}(\text{CO})_2\text{Cl}]_2$ , and  $[\text{Ir}(\text{CO})_3\text{Cl}]_n$  to give predominantly the mononuclear complexes  $[(\text{C}_5\text{H}_5\text{CH}_2\text{C}_5\text{H}_4)\text{M}(\text{CO})_2]$  (I;  $\text{M} = \text{Co}, \text{Rh}, \text{Ir}$ ) together with small amts. of the dinuclear compds.  $[\text{CH}_2(\text{C}_5\text{H}_4)_2][\text{M}(\text{CO})_2]_2$

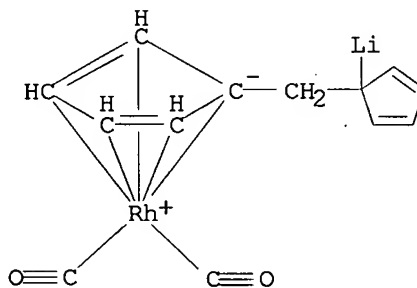


(II). The  $^1\text{H}$  and  $^{13}\text{C}$  NMR spectra of I prove that the  $\text{CH}_2\text{C}_5\text{H}_5$  substituent is linked to the  $\pi$ -bonded ring in two isomeric forms. Metalation of I ( $\text{M} = \text{Rh}, \text{Ir}$ ) with BuLi afford the lithiated derivs. from which on reaction with  $[\text{Co}(\text{CO})_4\text{I}]$ ,  $[\text{Rh}(\text{CO})_2\text{Cl}]_2$ , and  $[\text{C}_5\text{H}_5\text{TiCl}_3]$  the heteronuclear complexes  $[\text{CH}_2(\text{C}_5\text{H}_4)_2][\text{M}(\text{CO})_2][\text{M}'(\text{CO})_2]$  (III;  $\text{MM}' = \text{RhCo}, \text{IrCo}, \text{IrRh}$ ) and  $[\text{CH}_2(\text{C}_5\text{H}_4)_2][\text{Ir}(\text{CO})_2][\text{C}_5\text{H}_5\text{TiCl}_2]$  are obtained. Photolysis of III ( $\text{MM}' = \text{RhCo}, \text{IrCo}$ ) leads almost quant. to the formation of the CO-bridged compds.  $[\text{CH}_2(\text{C}_5\text{H}_4)_2][\text{M}(\text{CO})(\mu\text{-CO})\text{M}'(\text{CO})]$  (IV). According to an x-ray crystal structure anal. the Co/Rh complex IV ( $\text{MM}' = \text{RhCo}$ ) is isostructural to  $[\text{CH}_2(\text{C}_5\text{H}_4)_2][\text{Rh}_2(\text{CO})_2(\mu\text{-CO})]$ .

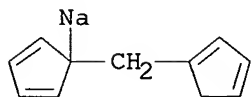
RX(2) OF 4



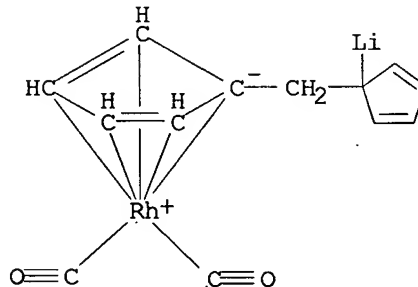
BuLi, Hexane →



RX(4) OF 4 - 2 STEPS



1.  $(\text{CO})_4\text{RhCl}_2\text{Rh}(\text{CO})_4$ ,  
THF  
2. BuLi, Hexane →



lithioboranyldiazomethane-lithioboranylisodiazomethane  
 [(>BCNN-,Li+)-( >BNNC-,Li+)] rearrangement

AUTHOR(S): Arthur, Marie Pierre; Baceiredo, Antoine; Bertrand, Guy

CORPORATE SOURCE: Lab. Chim. Coord., CNRS, Toulouse, 31077, Fr.

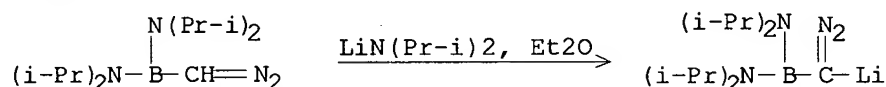
SOURCE: Journal of the American Chemical Society (1991), 113(15), 5856-7  
 CODEN: JACSAT; ISSN: 0002-7863

DOCUMENT TYPE: Journal

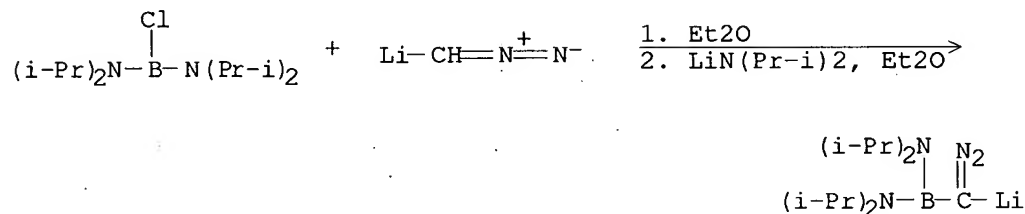
LANGUAGE: English

AB Bis(diisopropylamino)chloroborane reacts with the lithium salt of diazomethane, giving the first isolated  $\alpha$ -diazoborane [(Me<sub>2</sub>CH)<sub>2</sub>N] <sub>2</sub>BCHN<sub>2</sub> (I). Treatment of I with LDA in ether leads to lithio-boranyldiazomethane [(Me<sub>2</sub>CH)<sub>2</sub>N] <sub>2</sub>B-:C:N+:N-]Li<sup>+</sup> (II) whereas in THF the formation of [-C.tplbond.N+N:B-[N(CHMe<sub>2</sub>)<sub>2</sub>]<sub>2</sub>]Li<sup>+</sup> (III) is observed II can be rearranged irreversibly to III. Both II and III were characterized by IR and <sup>11</sup>B NMR spectroscopy, as well as by their chemical reactivity, which allows the synthesis of new stable nitrilimines. The exptl. results obtained for II and III are in marked contrast with the predictions of calcns. concerning the parent compds. HCNN- and CNNH-.

RX(2) OF 20



RX(8) OF 20 - 2 STEPS



L4 ANSWER 28 OF 50 CASREACT COPYRIGHT 2008 ACS on STN

ACCESSION NUMBER: 113:115452 CASREACT

TITLE: Reagents based on cyclopentadienyl derivatives of the Group 14 elements for the synthesis of indium(I) derivatives. Crystal and molecular structure of In(C<sub>5</sub>H<sub>4</sub>SiMe<sub>3</sub>)

AUTHOR(S): Beachley, O. T., Jr.; Lees, J. F.; Glassman, T. E.; Churchill, Melvyn Rowen; Buttrey, Lisa A.

CORPORATE SOURCE: Dep. Chem., State Univ. New York, Buffalo, NY, 14214, USA

SOURCE: Organometallics (1990), 9(9), 2488-92  
 CODEN: ORGND7; ISSN: 0276-7333

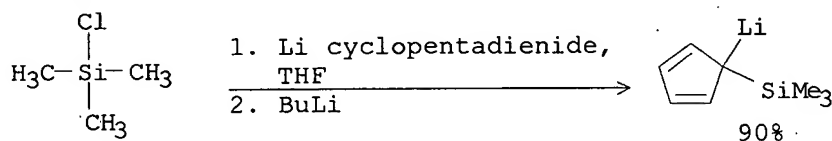
DOCUMENT TYPE: Journal

LANGUAGE: English

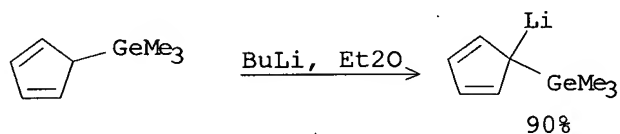
AB Cyclopentadienyl tri-Me derivs. of the Group 14 (Group IVA) elements (C<sub>5</sub>H<sub>4</sub>MMe<sub>3</sub>; M = Si, Ge, Sn) have been investigated for their effects on indium(I) chemical. The compds. In(C<sub>5</sub>H<sub>4</sub>SiMe<sub>3</sub>) (I) and In(C<sub>5</sub>H<sub>4</sub>GeMe<sub>3</sub>) have been prepared from the corresponding lithium cyclopentadienyl reagent LiC<sub>5</sub>H<sub>4</sub>MMe<sub>3</sub> and InCl. Characterization data have included partial elemental analyses (C, H), phys. properties, IR and <sup>1</sup>H NMR spectroscopic data, oxidation reactions with dilute aqueous HCl, and a single-crystal x-ray structural

study in the case of I. When  $C_5H_5SnMe_3$  was combined with  $InCl$  in  $Et_2O$ ,  $In(C_5H_5)$  and  $Me_3SnCl$  were formed in good yields. The solid-state structure of I consists of infinite zigzag chains of  $[In(C_5H_4SiMe_3)]_\infty$ . Each In atom interacts with 2  $\eta^5-C_5H_4SiMe_3$  ligands with a centroid...In...centroid angle of  $131.78^\circ$ , and each  $\eta^5-C_5H_4SiMe_3$  ligand is linked to 2 In atoms with In...centroid...In angles of  $175.94^\circ$ . There are no short interstrand In...In interactions, the shortest such distance being 5.428 Å. Thus,  $In(C_5H_4SiMe_3)$  is the 1st cyclopentadienylindium(I) derivative with no apparent indium-indium interactions.

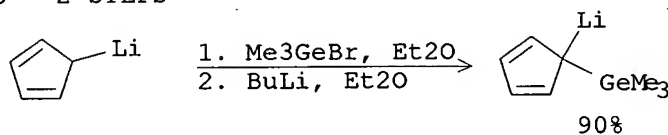
RX(1) OF 10



RX(4) OF 10



RX(8) OF 10 - 2 STEPS



L4 ANSWER 29 OF 50 CASREACT COPYRIGHT 2008 ACS on STN

ACCESSION NUMBER:

113:59481 CASREACT

TITLE:

Bis(cyclopentadienyl)methane bridged binuclear complexes. III. A new route to heteronuclear bimetallic complexes with  $[CH_2(C_5H_4)_2]^{2-}$  as bridged ligand

AUTHOR(S):

Schneider, D.; Werner, H.

CORPORATE SOURCE:

Inst. Anorg. Chem., Univ. Wuerzburg, Wuerzburg, D-8700, Germany

SOURCE:

Journal of Organometallic Chemistry (1990), 384(1-2), C33-C37

CODEN: JORCAI; ISSN: 0022-328X

DOCUMENT TYPE:

Journal

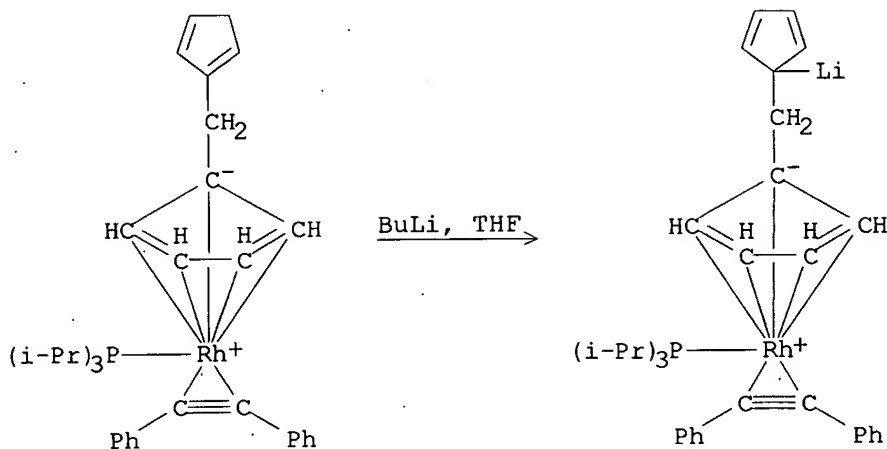
LANGUAGE:

German

AB

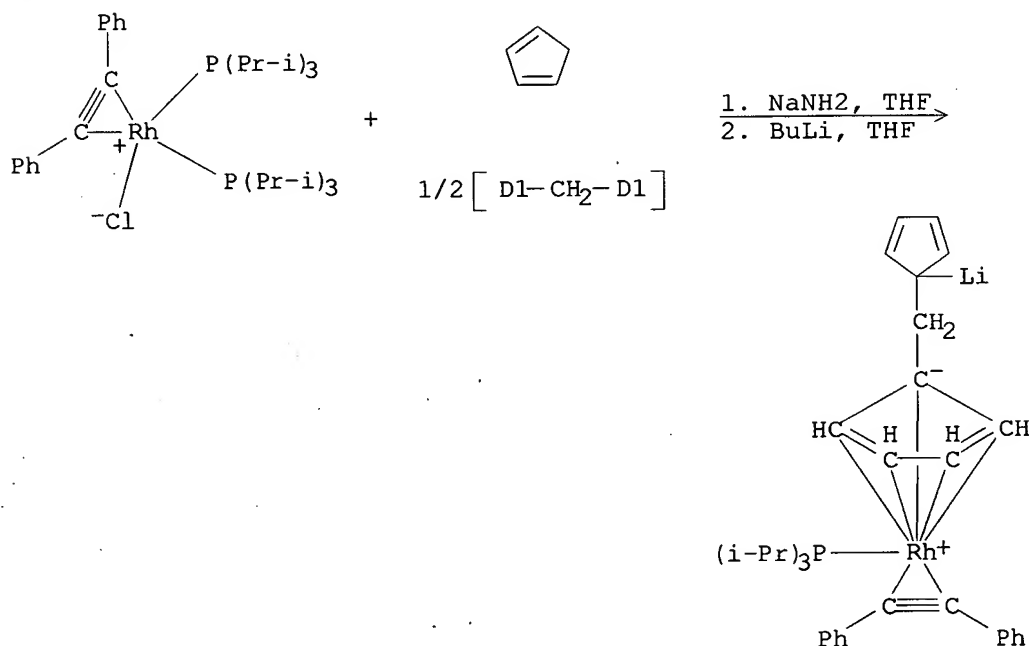
Reaction of the sodium salt  $NaC_5H_4R$  ( $R = CH_2C_5H_5$ ) which is obtained from  $CH_2(C_5H_5)_2$  and  $NaNH_2$  in THF with halo metal compds.  $[M(Hal)Lm]_n$  gives the complexes  $(C_5H_4R)ML_2$  in good yields. The  $^{13}C$  and  $^{31}P$  NMR spectra of these mononuclear compds. show that the substituent R is linked to the  $\pi$ -bonded ring in two isomeric forms. Metalation of  $(C_5H_4R)Rh(PhC.tplbond.CPh)[P(CHMe_2)_3]$  and  $(C_5H_4R)Ir(C_8H_{14})_2$  with  $n-BuLi$  in ether affords the lithiated derivs.  $(LiC_5H_4CH_2C_5H_4)ML_2$  which react with  $[Rh(CO)_2Cl]_2$  and  $[CoI(CO)_4]$  to give the heterodinuclear complexes. A second route to compds. generally formulated as  $(L_2M)(C_5H_4CH_2C_5H_4)(M'L'_2)$  which avoids the preparation of lithiated intermediates and uses  $trans-[Rh(C.tplbond.CMe)(py)[P(CHMe)_3]_2]$  as a substrate is also described.

RX(9) OF 25



RX(10) OF 25 - REACTION DIAGRAM NOT AVAILABLE

RX(16) OF 25 - 2 STEPS



NOTE: 1) 89% overall

RX(17) OF 25 - REACTION DIAGRAM NOT AVAILABLE

L4 ANSWER 30 OF 50 CASREACT COPYRIGHT 2008 ACS on STN

ACCESSION NUMBER: 112:35921 CASREACT

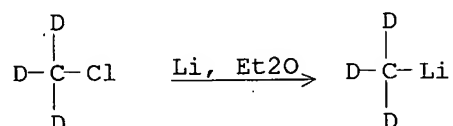
TITLE: Alkyl- and arylmetal compounds. XXXVII. Structure refinement of methylpotassium. Preparation of potassium trideuteriomethanide and neutron diffraction studies at 1.35 and 290 K

AUTHOR(S): Weiss, Erwin; Lambertsen, Thomas; Schubert, Bernd;

Cockcroft, Jeremy Karl  
 CORPORATE SOURCE: Inst. Anorg. Angew. Chem., Univ. Hamburg, Hamburg,  
 D-2000/13, Fed. Rep. Ger.  
 SOURCE: Journal of Organometallic Chemistry (1988), 358(1-3),  
 1-14  
 CODEN: JORCAI; ISSN: 0022-328X  
 DOCUMENT TYPE: Journal  
 LANGUAGE: German

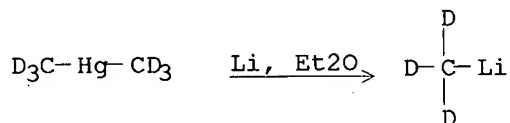
AB The crystal structure of methylpotassium has been refined by neutron diffraction carried out on powder samples of KCD3 at 1.35 and 290 K. Pyramidal Me ions were found with bond angles of 105° at 1.35 K and of 109° at 290 K, which are comparable to those of NH3. In the crystal the Me ions have alternating orientations and each carbanion is coordinated by six K ions in a distorted trigonal-prismatic array. The three K ions close to the sp3 lone electron pair have short K-C contacts (2 + 295, 1 + 302 pm), whereas the K ions close to the H atoms have longer K-C distances (2 + 344, 1 + 330 pm). An orthorhombic unit cell has now been found as compared to the smaller hexagonal cell (Z = 2) detected previously by x-ray methods (without precise location of the H atoms). The preparation of KCD3 and LiCD3 is described and IR data of KCD3 are given.

RX(2) OF 11



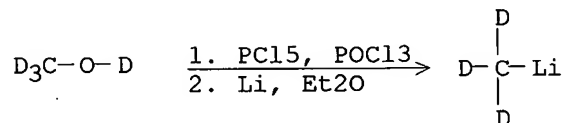
NOTE: The product contains chloride ions

RX(3) OF 11



NOTE: The product is chloride free

RX(6) OF 11 - 2 STEPS



NOTE: 2) The product contains chloride ions

L4 ANSWER 31 OF 50 CASREACT COPYRIGHT 2008 ACS on STN

ACCESSION NUMBER: 111:233121 CASREACT

TITLE: Synthesis, x-ray structure, and reactivity with Lewis acids of metallacyclopentane derivatives of rhodium(III) and iridium(III). Observation of the first boron trifluoride-promoted carbon-hydrogen bond activation in transition metal alkyls

AUTHOR(S): Bertani, Roberto; Diversi, Pietro; Ingrosso, Giovanni; Lucherini, Antonio; Marchetti, Fabio; Adovasio, Victor; Nardelli, Mario

CORPORATE SOURCE: Dip. Chim. Chim. Ind., Univ. Pisa, Pisa, 56100, Italy

SOURCE:

Journal of the Chemical Society, Dalton Transactions:  
Inorganic Chemistry (1972-1999) (1988), (12), 2983-94  
CODEN: JCDTBI; ISSN: 0300-9246

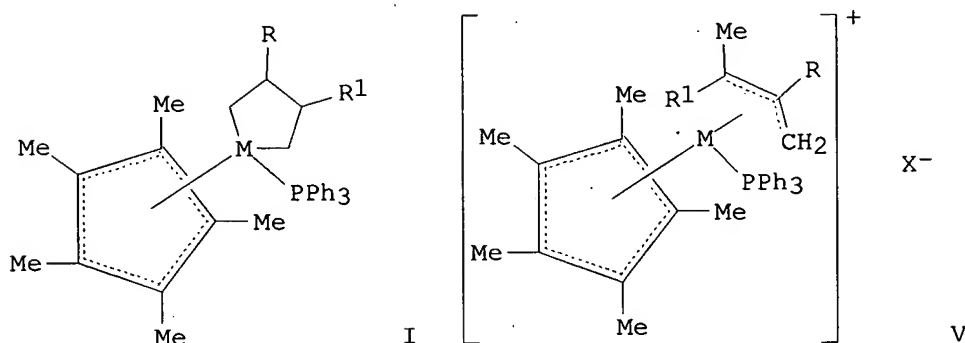
DOCUMENT TYPE:

Journal

LANGUAGE:

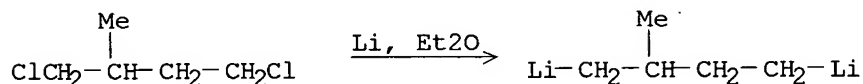
English

GI



AB Metallacyclopentanes I [R = Me, R1 = H, M = Rh, Ir (II); R = R1 = Me, M = Rh (III), Ir (IV); R = Me2CH, R1 = H, M = Ir] were prepared by treating M1CH2CHRCHR1CH2M1 (M1 = Li, MgCl) with MCl2( $\eta^5$ -C5Me5)(PPh3) (C5H5 = cyclopentadienyl). The x-ray crystal structures of II-IV were determined. All compds. possess a 3-legged piano stool structure, the major differences lying in the puckering of the metallocyclopentane rings. The metallacyclopentanes react under mild conditions with BF3·Bu2O and with [Ph3C]PF6 to give  $\eta^3$ -allyl complexes V [R = R1 = H, M = Rh, Ir; R = Me, R1 = H, M = Rh, Ir; R = R1 = Me, M = Rh, Ir; X = BF4, PF6], via regiospecific hydrogen abstraction from the alkyl-substituted  $\beta$ -carbon atom by Lewis acids. Complexes V were characterized by elemental anal., <sup>1</sup>H NMR, and fast-atom-bombardment mass spectrometry.

RX(18) OF 42



L4 ANSWER 32 OF 50 CASREACT COPYRIGHT 2008 ACS on STN

ACCESSION NUMBER: 110:135444 CASREACT

TITLE: A convenient synthesis of C5(CD3)5H. Synthesis and characterization of Fe( $\eta^5$ -C5(CD3)5)2

AUTHOR(S): O'Hare, Dermot; Manriquez, Juan; Miller, Joel S.

CORPORATE SOURCE: Cent. Res. Devl. Dep., E. I. du Pont de Nemours and Co., Inc., Wilmington, DE, 19898, USA

SOURCE: Journal of the Chemical Society, Chemical Communications (1988), (7), 491-3  
CODEN: JCCCAT; ISSN: 0022-4936

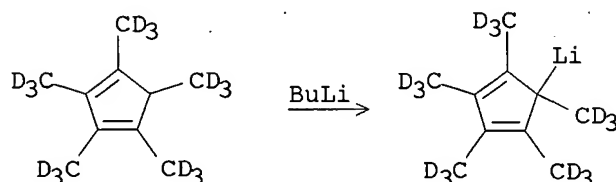
DOCUMENT TYPE: Journal

LANGUAGE: English

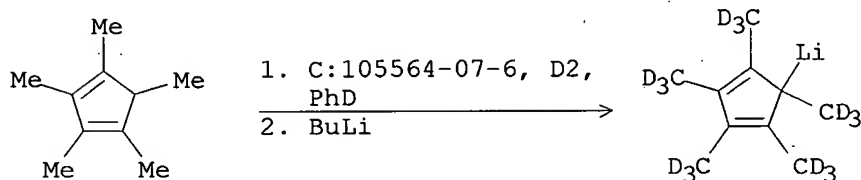
AB The large-scale preparation of deuterio(pentamethylcyclopentadiene), C5(CD3)5H ([2H15]Cp\*H), from C5Me5H with C6D6, and subsequent synthesis of

[2H30][Fe( $\eta$ -Cp\*)2] is described, together with IR, Raman, and solid state 2H NMR spectroscopic characterization.

RX(2) OF 4



RX(4) OF 4 - 2 STEPS



L4 ANSWER 33 OF 50 CASREACT COPYRIGHT 2008 ACS on STN

ACCESSION NUMBER: 110:77481 CASREACT

TITLE: Diphenylmethane and triphenylmethane dye ethynovinylogues with absorption bands in the near-infrared

AUTHOR(S): Akiyama, Shuzo; Nakatsuji, Shinichi; Nakashima, Kenichiro; Yamasaki, Seiko

CORPORATE SOURCE: Fac. Pharm. Sci., Nagasaki Univ., Nagasaki, 852, Japan

SOURCE: Dyes and Pigments (1988), 9(6), 459-66

CODEN: DYPIDX; ISSN: 0143-7208

DOCUMENT TYPE: Journal

LANGUAGE: English

AB Diphenylmethane and triphenylmethane dye ethynovinylogs (i.e., substituted (ethynyl) (vinyl) carbenium ions), light-absorbing in the near-IR (up to 814 nm in CH<sub>2</sub>Cl<sub>2</sub>), were synthesized by treatment of 1,3,5-trisubstituted 1-penten-4-yn-3-ols with acids. The light absorption of (p-dimethylaminophenyl) (p-dimethylaminophenylethynyl) (phenyl)carbenium perchlorate and related dyes in acidic medium is described.

RX(4) OF 12



L4 ANSWER 34 OF 50 CASREACT COPYRIGHT 2008 ACS on STN

ACCESSION NUMBER: 109:231188 CASREACT

TITLE: Decaphenylgermanocene, -stannocene and -plumbocene and pentaphenylstannocene: synthesis properties and CPMAS-metal-NMR measurements

AUTHOR(S): Janiak, Christoph; Schumann, Herbert; Stader, Carin; Wrackmeyer, Bernd; Zuckerman, Jerold J.

CORPORATE SOURCE: Inst. Anorg. Anal. Chem., Tech. Univ. Berlin, Berlin, D-1000/12, Fed. Rep. Ger.

SOURCE: Chemische Berichte (1988), 121(10), 1745-51

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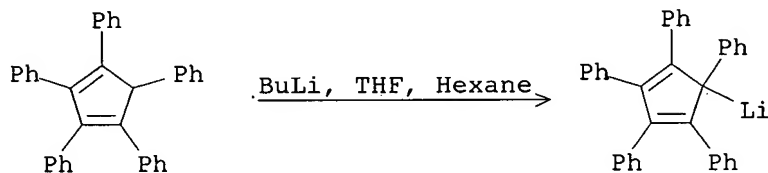
Journal

LANGUAGE:

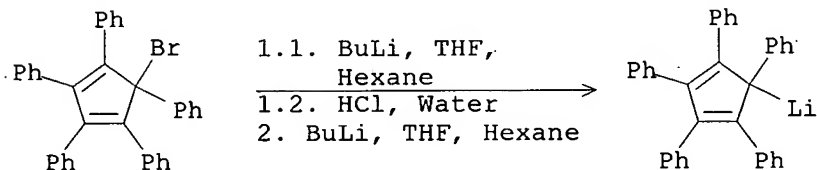
German

AB Reaction of tetraphenylcyclopentadienone with PhLi, followed by acidic hydrolysis gave 83% pentaphenylcyclopentadienol which on bromination with HBr-H<sub>2</sub>O followed by lithiation and acidic hydrolysis gave 1,2,3,4,5-pentaphenyl-1,3-cyclopentadiene (I). The reaction of the lithium or sodium salts of I with Group IV metal dihalides gave title compds., ( $\eta^5$ -C<sub>5</sub>Ph<sub>5</sub>)<sub>2</sub>M (II; M = Ge, Sn, Pb) complete anal. data (IR, Raman, x-ray-powder, NMR, mass spectra, and <sup>119</sup>mSn Moessbauer spectra) of II are given. <sup>119</sup>Sn and <sup>207</sup>Pb CPMAS-NMR of II (M = Sn, Pb) are reported for the first time.

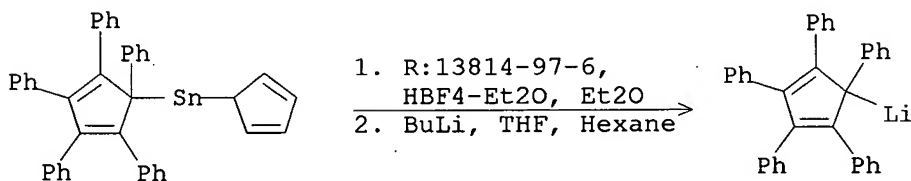
RX(4) OF 30



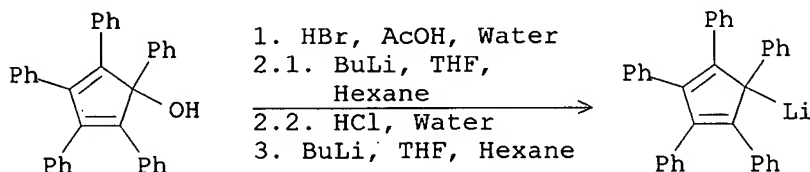
RX(12) OF 30 - 2 STEPS



RX(13) OF 30 - 2 STEPS

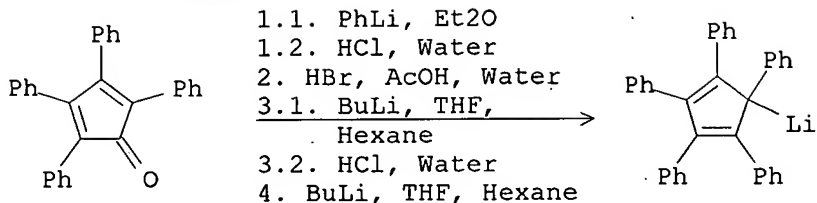


RX(18) OF 30 - 3 STEPS

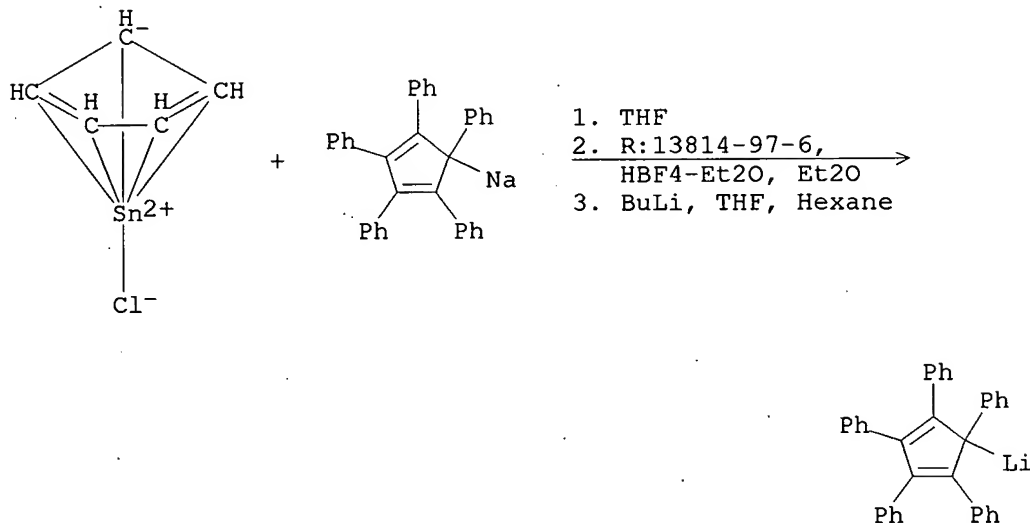




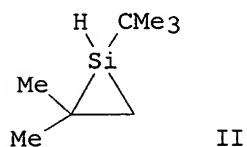
RX(19) OF 30 - 4 STEPS



RX(28) OF 30 - 3 STEPS



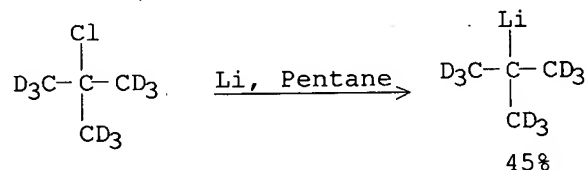
L4 ANSWER 35 OF 50 CASREACT COPYRIGHT 2008 ACS on STN  
 ACCESSION NUMBER: 109:129120 CASREACT  
 TITLE: The photochemistry of matrix-isolated di-tert-butylidiazidosilane. Observation of di-tert-butylsilylene and N,N'-di-tert-butylsilanediimine  
 AUTHOR(S): Welsh, Kevin M.; Michl, Josef; West, Robert  
 CORPORATE SOURCE: Dep. Chem., Univ. Wisconsin, Madison, WI, 53706, USA  
 SOURCE: Journal of the American Chemical Society (1988), 110(20), 6689-96  
 CODEN: JACSAT; ISSN: 0002-7863  
 DOCUMENT TYPE: Journal  
 LANGUAGE: English  
 GI



AB The major product from 254-nm irradiation of matrix-isolated (Me<sub>3</sub>C)<sub>2</sub>Si(N<sub>3</sub>)<sub>2</sub> is (Me<sub>3</sub>C)<sub>2</sub>Si: (I), a highly reactive ground-state singlet species with

$\lambda_{\text{max}}$  480 nm, which undergoes a subsequent photochem. C-H insertion to give the stable silacyclopropane II. The photochem. formation of I proceeds in at least two steps. A small amount (<5%) of a photochem. precursor to I is observed, with  $\lambda_{\text{max}}$  at 300 nm and an IR band at 2150  $\text{cm}^{-1}$ , tentatively assigned as  $(\text{Me}_3\text{C})_2\text{Si}:\text{N}_2$ . The irradiation of  $(\text{Me}_3\text{C})_2\text{Si}(\text{N}_3)_2$  also yields  $\text{Me}_3\text{CN}:\text{Si}:\text{NCMe}_3$  as a minor (11%) product, with  $\lambda_{\text{max}}$  240 and 385 nm. This process also involves at least two steps, and a very small yield of an intermediate ( $\lambda_{\text{max}}$ , 725 nm) of an unknown structure was detected.

RX(4) OF 32



L4. ANSWER 36 OF 50 CASREACT COPYRIGHT 2008 ACS on STN

ACCESSION NUMBER: 109:116944 CASREACT

TITLE: Regeneration of catalysts containing platinum-group metals

INVENTOR(S): Jegorov, Alexandr; Podlaha, Jaroslav; Polievka, Milan

PATENT ASSIGNEE(S): Czech.

SOURCE: Czech., 5 pp.

CODEN: CZXXA9

DOCUMENT TYPE: Patent

LANGUAGE: Czech

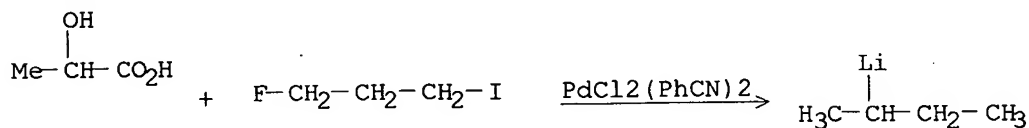
FAMILY ACC. NUM. COUNT: 1

PATENT INFORMATION:

PATENT NO.	KIND	DATE	APPLICATION NO.	DATE
CS 246734	B1	19861113	CS 1984-7639	19841009
PRIORITY APPLN. INFO.:			CS 1984-7639	19841009

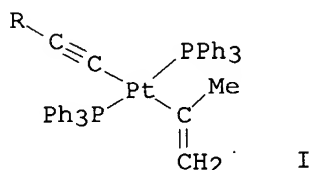
AB Catalytically active metals, including Pd, Os, Rh, Pt, Ir, and Ru, give with phosphines, containing a hydrophilic group, H<sub>2</sub>O-soluble complexes which are readily separated from the organic reaction components. The process is applicable to numerous isomerization, hydrosilylation, hydroformylation, hydrogenation, and oxidation catalysts. A spent  $\text{PdCl}_2(\text{PhCN})_2$  catalyst, after 1-pentene isomerization in  $\text{C}_6\text{H}_6$ , was treated with  $[(\text{NaO}_2\text{CCH}_2)_2\text{PCH}_2]_2$  to obtain a P/Pd ratio of 4; the mixture was vigorously stirred with H<sub>2</sub>O, and allowed to sep. to yield 96.6% Pd in the aqueous phase and 10.9 ppm. Pd in the  $\text{C}_6\text{H}_6$  phase.

RX(2) OF 2



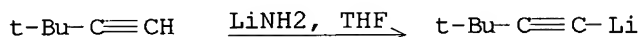
Na

ACCESSION NUMBER: 108:204828 CASREACT  
 TITLE: Mixed  $\sigma$ -alkynyl- $\sigma$ -vinyl  
 bis(triphenylphosphine)platinum complexes via a labile  
 $\sigma$ -vinyl platinum triflate precursor  
 AUTHOR(S): Kowalski, Mark H.; Arif, Atta M.; Stang, Peter J.  
 CORPORATE SOURCE: Dep. Chem., Univ. Utah, Salt Lake City, UT, 84112, USA  
 SOURCE: Organometallics (1988), 7(5), 1227-9  
 CODEN: ORGND7; ISSN: 0276-7333  
 DOCUMENT TYPE: Journal  
 LANGUAGE: English  
 GI

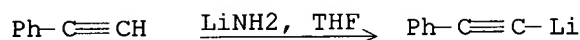


AB Addition of a vinyl platinum(II) complex with a labile triflate ligand to a THF solution of selected acetylides  $\text{LiC}\equiv\text{C}\cdot\text{R}$  ( $\text{R} = \text{CMe}_3, \text{Ph}, \text{CH}_2\text{OMe}$ ), generated from the corresponding terminal acetylenes and  $\text{LiNH}_2$ , gives trans- $\sigma$ -alkynyl- $\sigma$ -vinyl Pt(II) complexes I in good yields. NMR and IR data and x-ray structure determination for I ( $\text{R} = \text{Me}_3\text{C}$ ) are described.

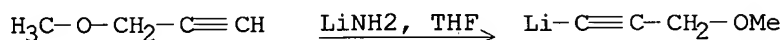
RX(1) OF 16



RX(2) OF 16



RX(3) OF 16



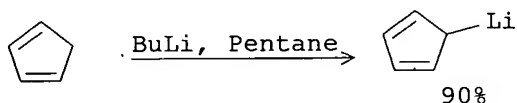
ACCESSION NUMBER: 108:204671 CASREACT  
 TITLE: Synthesis, characterization and structural studies of  
 $\text{In}(\text{C}_5\text{H}_4\text{Me})$  by x-ray diffraction and electron  
 diffraction techniques and a reinvestigation of the  
 crystalline state of  $\text{In}(\text{C}_5\text{H}_5)$  by x-ray diffraction  
 studies  
 AUTHOR(S): Beachley, O. T., Jr.; Pazik, J. C.; Glassman, T. E.;  
 Churchill, Melvyn Rowen; Fettingner, James C.; Blom,  
 Richard  
 CORPORATE SOURCE: Dep. Chem., State Univ. New York, Buffalo, NY, 14214,  
 USA  
 SOURCE: Organometallics (1988), 7(5), 1051-9  
 CODEN: ORGND7; ISSN: 0276-7333

DOCUMENT TYPE: Journal

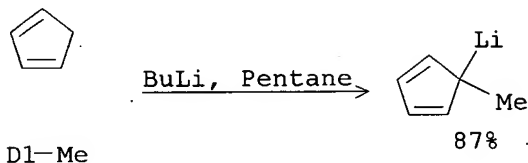
LANGUAGE: English

AB The compound  $\text{In}(\text{C}_5\text{H}_4\text{Me})$  was prepared from  $\text{InCl}$  and  $\text{Li}(\text{C}_5\text{H}_4\text{Me})$  in  $\text{Et}_2\text{O}$  and fully characterized according to its phys. and solubility properties, reaction with dilute  $\text{HCl}$ , a cryoscopic mol. weight study in cyclohexane, IR and  $^1\text{H}$  NMR spectroscopic properties, an x-ray structural study, and a gas-phase electron diffraction study. In addition, a quant. x-ray structural study was used to reinvestigate the nature of the solid state of  $\text{In}(\text{C}_5\text{H}_5)$ . Each structure consists of zigzag chains of  $\text{InCp}$  units ( $\text{Cp} = \text{C}_5\text{H}_5$  or  $\text{C}_5\text{H}_4\text{Me}$ ) in which  $\text{In}$  atoms interact with each side of the  $\text{Cp}$  ring and two  $\text{Cp}$  rings interact with each  $\text{In}$  atom. Weak interchain  $\text{In-In}$  interactions at 3.986 (1) Å are observed for both  $\text{In}(\text{C}_5\text{H}_5)$  and  $\text{In}(\text{C}_5\text{H}_4\text{Me})$ , although they crystallize in different space groups. The mol. structure of  $\text{In}(\text{C}_5\text{H}_4\text{Me})$  in the gas-phase consists of discrete monomeric units with the  $\text{In(I)}$  atom being situated above the ring centroid. The available data permit comparisons of the properties and structural parameters of  $\text{In}(\text{C}_5\text{H}_5)$ ,  $\text{In}(\text{C}_5\text{H}_4\text{Me})$ , and  $\text{In}(\text{C}_5\text{Me}_5)$ .

RX(1) OF 6



RX(2) OF 6



L4 ANSWER 39 OF 50 CASREACT COPYRIGHT 2008 ACS on STN

ACCESSION NUMBER: 108:94687 CASREACT

TITLE: Tetraphenylcyclopentadiene and (4-tert-butylphenyl)tetraphenylcyclopentadiene: synthesis and characterization of their alkali-metal salts and metallocenes of germanium, tin, and lead  
AUTHOR(S): Schumann, Herbert; Janiak, Christoph; Zuckerman, Jerold J.

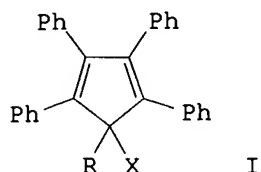
CORPORATE SOURCE: Inst. Anorg. Anal. Chem., Tech. Univ. Berlin, Berlin, D-1000/12, Fed. Rep. Ger.

SOURCE: Chemische Berichte (1988), 121(2), 207-18  
CODEN: CHBEAM; ISSN: 0009-2940

DOCUMENT TYPE: Journal

LANGUAGE: German

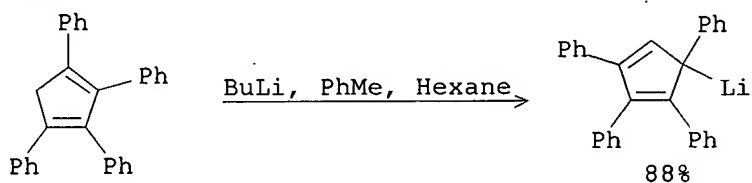
GI



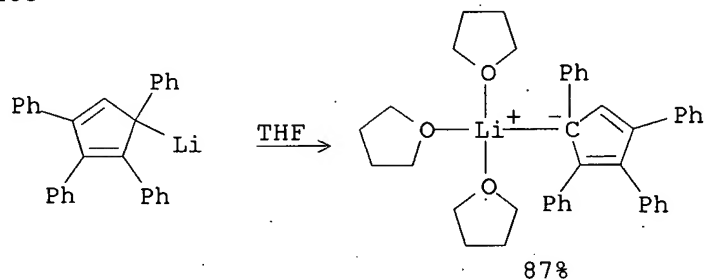
AB Title compds. I ( $\text{R} = \text{H}, \text{Ph}, 4\text{-Me}_3\text{CC}_6\text{H}_4$ ;  $\text{X} = \text{H}, \text{Li}, \text{Na}, \text{K}, \text{Br}, \text{OH}$ ) and the

Ge, Sn, and Pb metallocenes of I ( $R = H, 4-Me_3CC_6H_4$ ;  $X = H$ ) were prepared IR, Raman,  $^1H$  and  $^{13}C$  NMR and mass spectral data and x-ray powder diagrams were reported. Comparative  $^{13}C$  NMR studies showed delocalization of the neg. charge from the cyclopentadiene ring into the nonparallel Ph ligands.

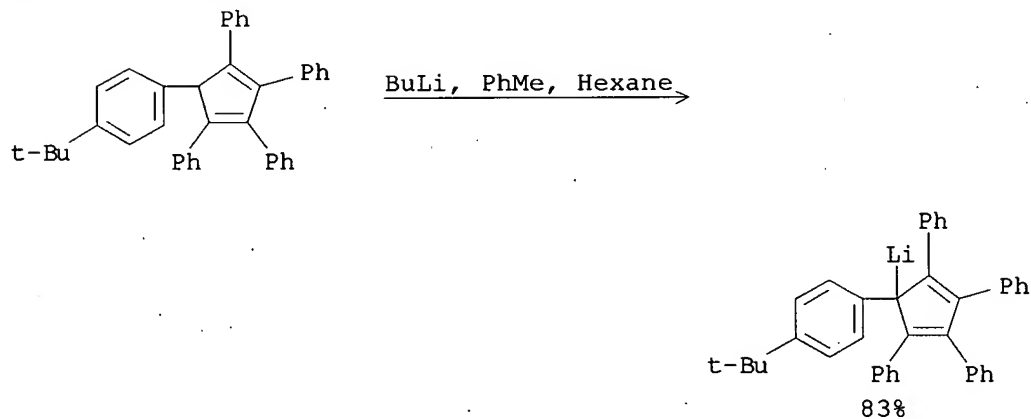
RX(1) OF 185



RX(2) OF 185

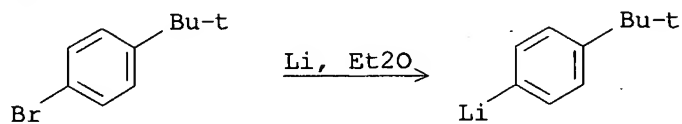


RX(12) OF 185

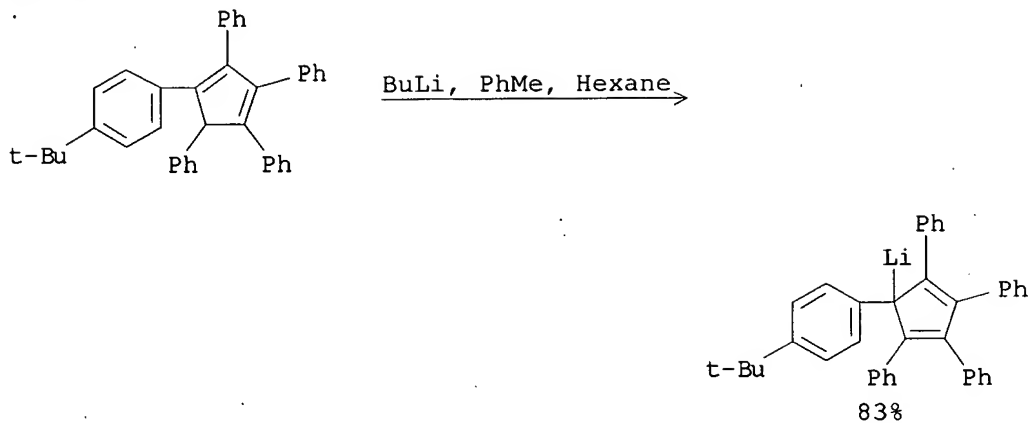


NOTE: ISOMERIC REACTANT ALSO PRESENT

RX(22) OF 185

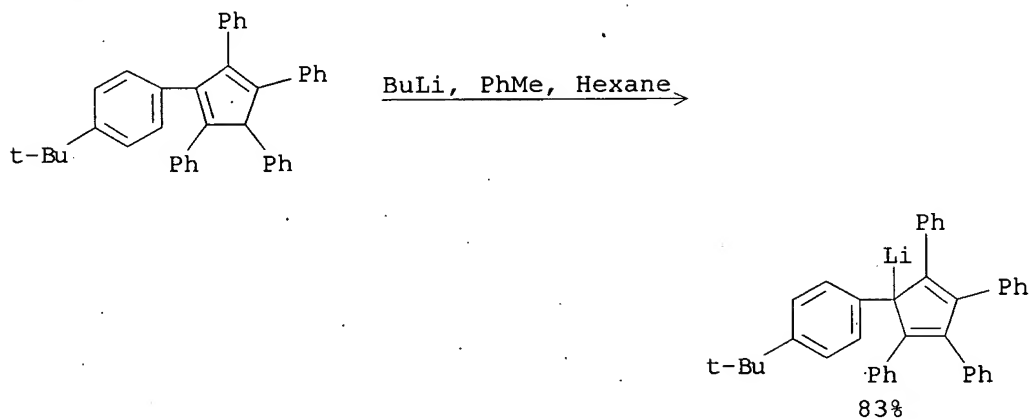


RX(25) OF 185



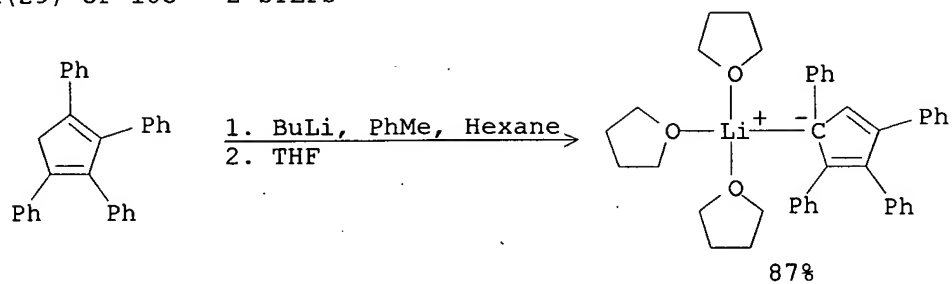
NOTE: ISOMERIC REACTANT ALSO PRESENT

RX(26) OF 185

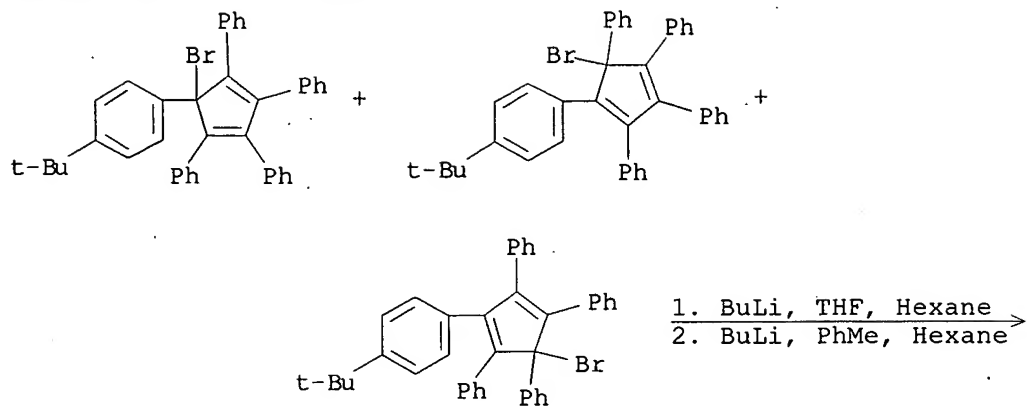


NOTE: ISOMERIC REACTANT ALSO PRESENT

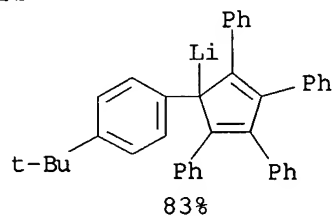
RX(29) OF 185 - 2 STEPS



RX(38) OF 185 - 2 STEPS

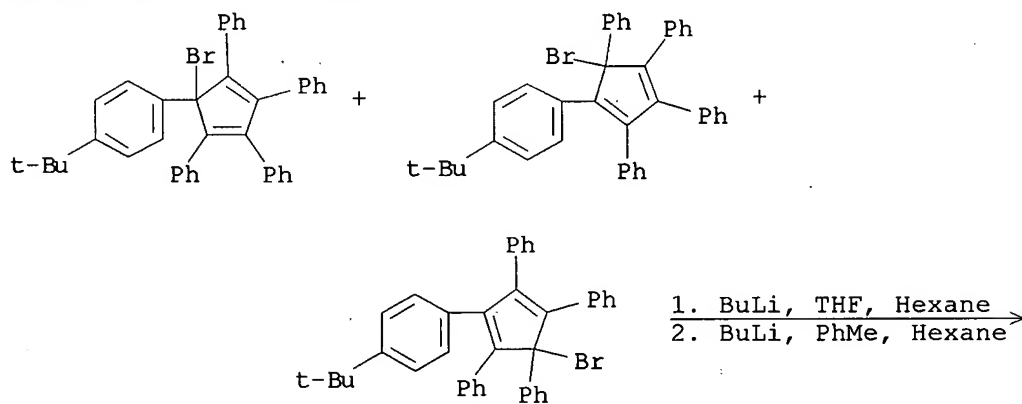


RX(38) OF 185 - 2 STEPS

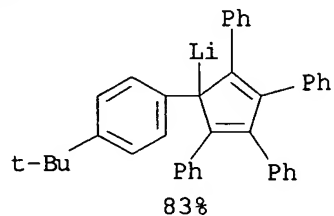


NOTE: 1) 93% overall, 2) ISOMERIC REACTANT ALSO PRESENT

RX(40) OF 185 - 2 STEPS

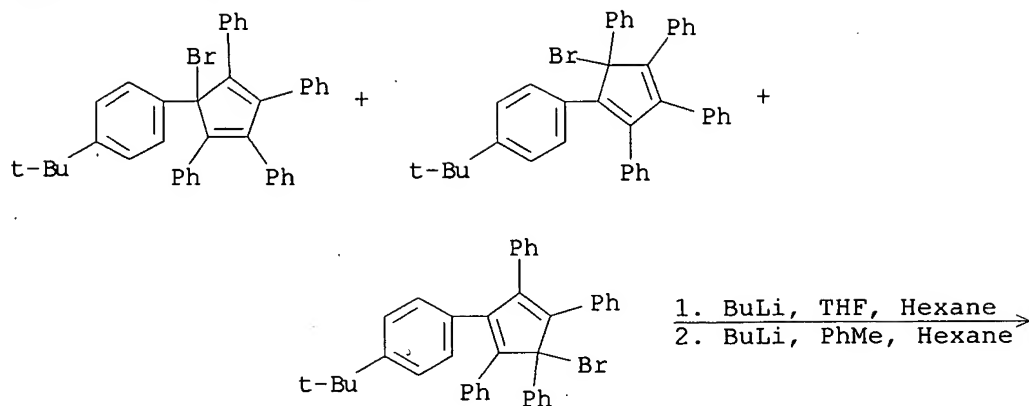


RX(40) OF 185 - 2 STEPS

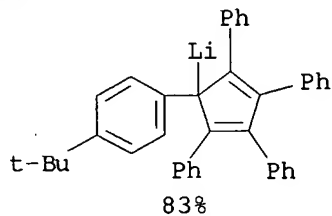


NOTE: 1) 93% overall, 2) ISOMERIC REACTANT ALSO PRESENT

RX(42) OF 185 - 2 STEPS

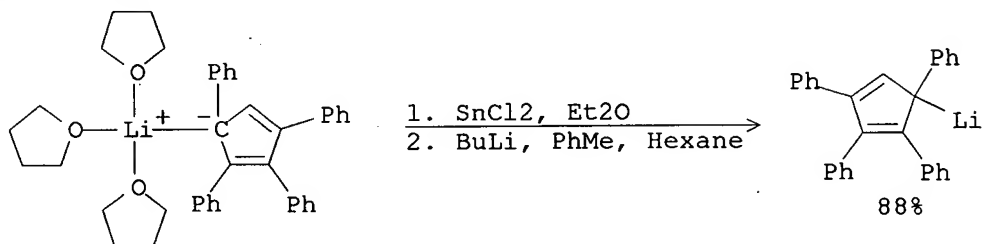


RX(42) OF 185 - 2 STEPS

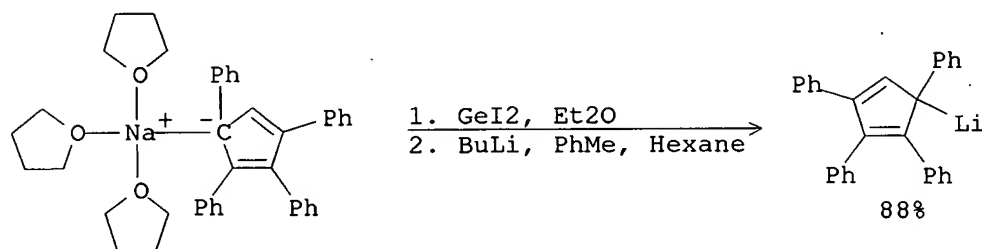


NOTE: 1) 93% overall, 2) ISOMERIC REACTANT ALSO PRESENT

RX(62) OF 185 - 2 STEPS

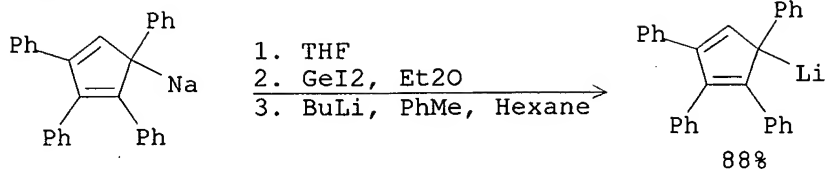


RX(66) OF 185 - 2 STEPS

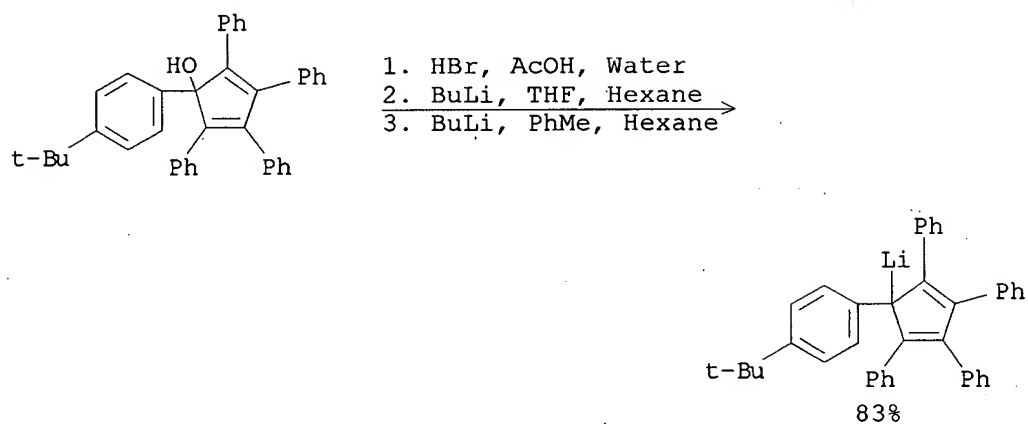




RX(74) OF 185 - 3 STEPS

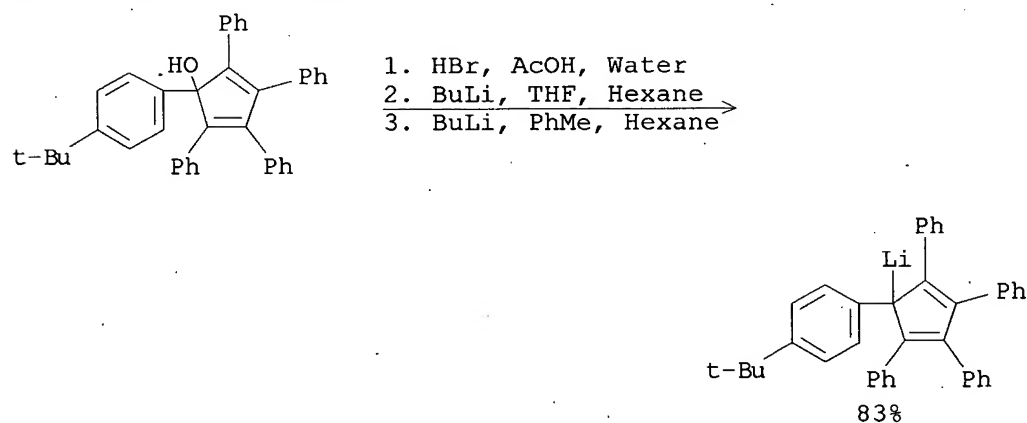


RX(76) OF 185 - 3 STEPS



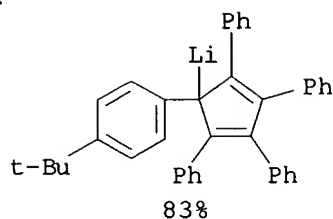
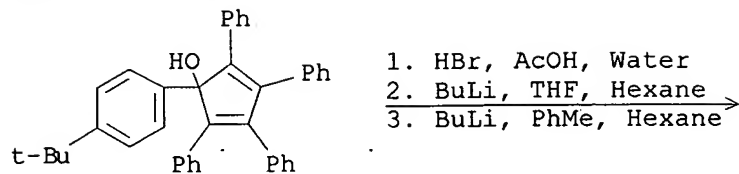
NOTE: 1) 73% overall, 2) 93% overall, 3) ISOMERIC REACTANT ALSO PRESENT

RX(78) OF 185 - 3 STEPS



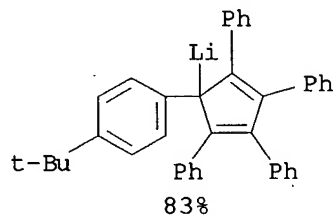
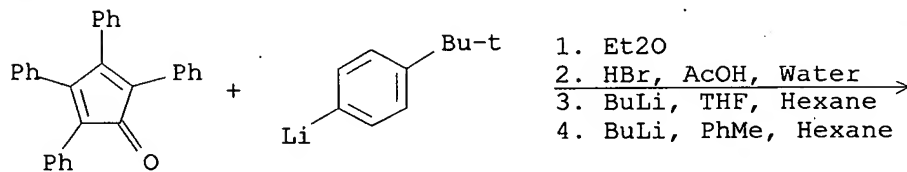
NOTE: 1) 73% overall, 2) 93% overall, 3) ISOMERIC REACTANT ALSO PRESENT

RX(80) OF 185 - 3 STEPS



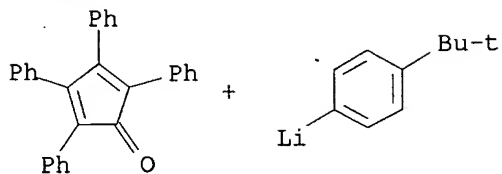
NOTE: 1) 73% overall, 2) 93% overall, 3) ISOMERIC REACTANT ALSO PRESENT

RX(82) OF 185 - 4 STEPS

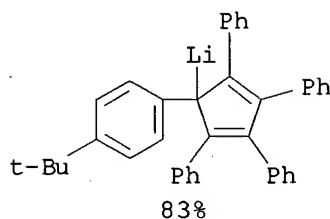


NOTE: 2) 73% overall, 3) 93% overall, 4) ISOMERIC REACTANT ALSO PRESENT

RX(84) OF 185 - 4 STEPS



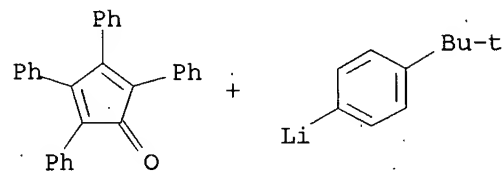
1. Et<sub>2</sub>O
2. HBr, AcOH, Water
3. BuLi, THF, Hexane
4. BuLi, PhMe, Hexane



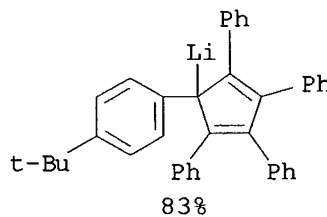
83%

NOTE: 2) 73% overall, 3) 93% overall, 4) ISOMERIC REACTANT ALSO PRESENT

RX(86) OF 185 - 4 STEPS



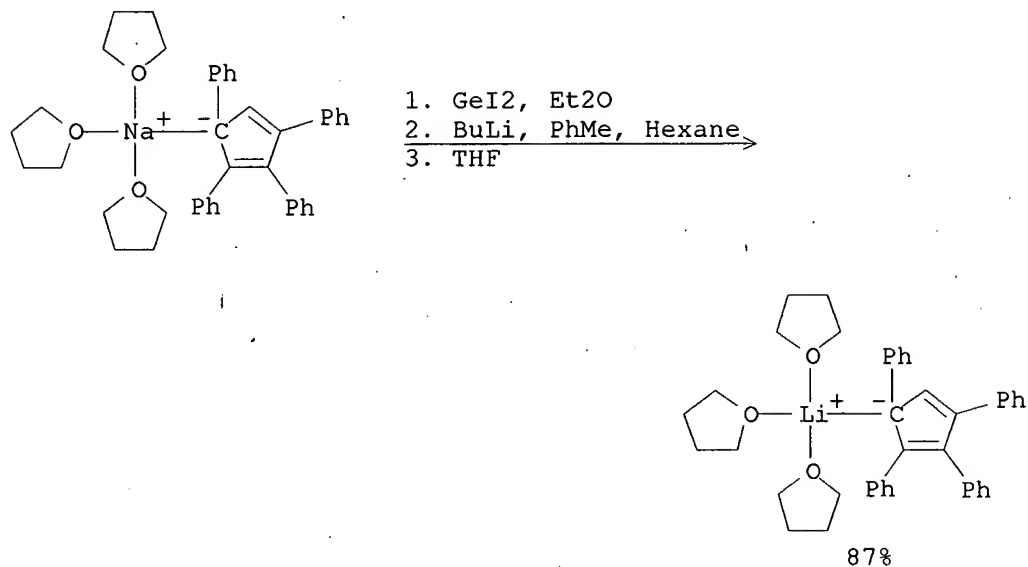
1. Et<sub>2</sub>O
2. HBr, AcOH, Water
3. BuLi, THF, Hexane
4. BuLi, PhMe, Hexane



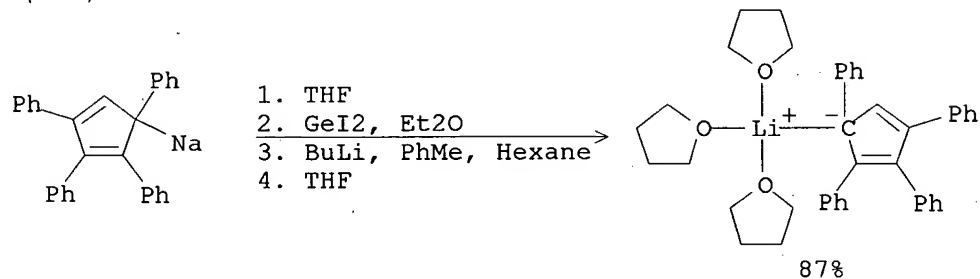
83%

NOTE: 2) 73% overall, 3) 93% overall, 4) ISOMERIC REACTANT ALSO PRESENT

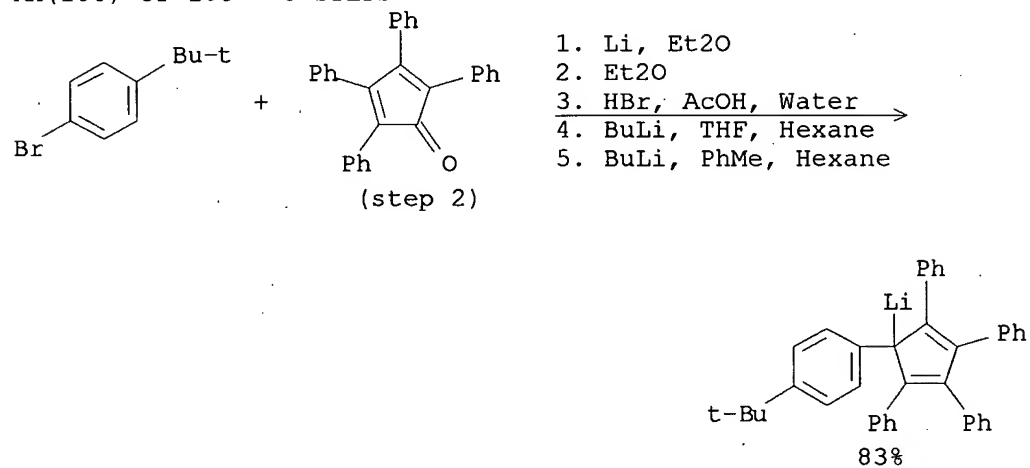
RX(129) OF 185 - 3 STEPS



RX(136) OF 185 - 4 STEPS

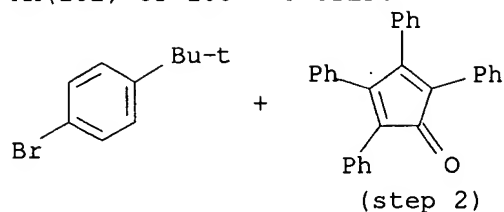


RX(180) OF 185 - 5 STEPS

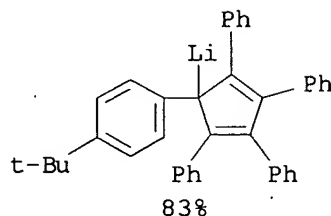


NOTE: 3) 73% overall, 4) 93% overall, 5) ISOMERIC REACTANT ALSO PRESENT

RX(182) OF 185 - 5 STEPS

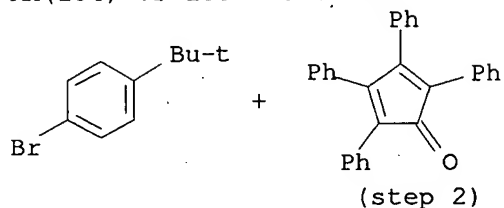


1. Li, Et<sub>2</sub>O
2. Et<sub>2</sub>O
3. HBr, AcOH, Water
4. BuLi, THF, Hexane
5. BuLi, PhMe, Hexane

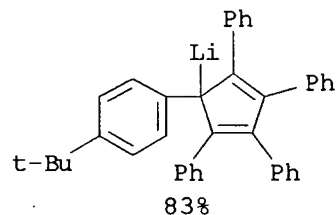


NOTE: 3) 73% overall, 4) 93% overall, 5) ISOMERIC REACTANT ALSO PRESENT

RX(184) OF 185 - 5 STEPS



1. Li, Et<sub>2</sub>O
2. Et<sub>2</sub>O
3. HBr, AcOH, Water
4. BuLi, THF, Hexane
5. BuLi, PhMe, Hexane



NOTE: 3) 73% overall, 4) 93% overall, 5) ISOMERIC REACTANT ALSO PRESENT

L4 ANSWER 40 OF 50 CASREACT COPYRIGHT 2008 ACS on STN

ACCESSION NUMBER: 107:40034 CASREACT

TITLE: Photochemistry of  $\eta^4$ -cyclopentadiene iron tricarbonyl complexes: transfer of the 5-endo substituent to the iron center following dissociative loss of carbon monoxide

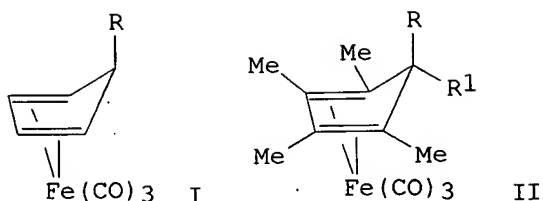
AUTHOR(S): Zou, Chaofeng; Wrighton, Mark S.; Blaha, Josephine Paw  
CORPORATE SOURCE: Dep. Chem., Massachusetts Inst. Technol., Cambridge, MA, 02139, USA

SOURCE: Organometallics (1987), 6(7), 1452-8  
CODEN: ORGND7; ISSN: 0276-7333

DOCUMENT TYPE: Journal

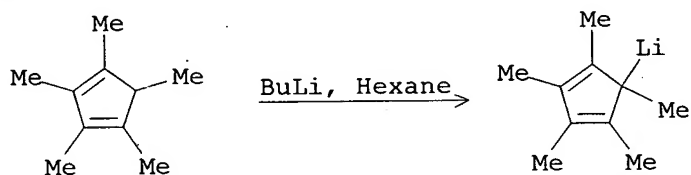
LANGUAGE: English

GI



AB Near-UV irradiation of  $(\eta^4\text{-C}_5\text{H}_5\text{R})\text{Fe}(\text{CO})_3$  (I, R = *exo*-H, *exo*-CH<sub>2</sub>Ph) and  $(\eta^4\text{-C}_5\text{Me}_4\text{RR}_1)\text{Fe}(\text{CO})_3$  (R = *exo*-Me, R<sub>1</sub> = *endo*-Me; R = *exo*-Me, R<sub>1</sub> = *endo*-H; R = *exo*-CH<sub>2</sub>Ph; R<sub>1</sub> = *endo*-Me) results in the dissociative loss of CO to give coordinatively unsatd. 16e photoproduct  $(\eta^4\text{-cyclopentadiene})\text{Fe}(\text{CO})_2$  that can be detected by IR at low temperature, .apprx.77 K. Irradiation ( $\lambda > 420$  nm) of the 16e species at low temperature (.apprx.77 K) results in transfer of the 5-*endo* substituent to form  $(\eta^5\text{-cyclopentadienyl})\text{Fe}(\text{CO})_2\text{R}_1$  (R<sub>1</sub> = H, Me) products. Transfer of the 5-*endo* substituent can also occur thermally upon warming, but the transfer of the 5-*endo* group is not very competitive with back-reaction of the 16e species and CO to regenerate the starting  $(\eta^4\text{-cyclopentadiene})\text{Fe}(\text{CO})_3$  complexes. The 366-nm quantum yield (at 10<sup>-7</sup> einstein/min) for CO loss at 298 K in alkane solution is .apprx.0.1; the formation quantum yields for the  $(\eta^5\text{-cyclopentadienyl})\text{Fe}(\text{CO})_2\text{H}$  species are also .apprx.0.1, but the formation quantum yields for the  $(\eta^5\text{-cyclopentadienyl})\text{Fe}(\text{CO})_2\text{Me}$  species are <10<sup>-2</sup>. All results are consistent with thermal or light-activated transfer of the 5-*endo* substituent (H or Me) following light-induced loss of CO from the parent  $(\eta^4\text{-cyclopentadiene})\text{Fe}(\text{CO})_3$ . The thermal rate of the transfer of a 5-*endo*-Me vs. a 5-*endo*-H is much slower on the basis of the thermal rates of reactions of  $(\eta^4\text{-C}_5\text{Me}_5\text{R}_1)\text{Fe}(\text{CO})_2$  (1-pentene) (R<sub>1</sub> = *endo*-H or *endo*-Me) which give  $(\eta^5\text{-C}_5\text{Me}_5)\text{Fe}(\text{CO})_2\text{R}_1$ .

RX(5) OF 10



L4 ANSWER 41 OF 50 CASREACT COPYRIGHT 2008 ACS on STN

ACCESSION NUMBER: 107:7371 CASREACT

TITLE: The chemistry of bulky chelating phosphines. 3. Anionic alkyl and aryl complexes of rhodium(I) and iridium(I)

AUTHOR(S): Del Paggio, Alan A.; Andersen, Richard A.; Muetterties, Earl L.

CORPORATE SOURCE: Dep. Chem., Univ. California, Berkeley, CA, 94720, USA

SOURCE: Organometallics (1987), 6(6), 1260-7

CODEN: ORGND7; ISSN: 0276-7333

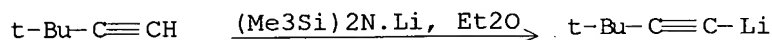
DOCUMENT TYPE: Journal

LANGUAGE: English

AB A series of thermally stable dialkyl-, diaryl-, and dialkynylrhodium(I) and -iridium(I) bis(phosphine) complexes  $(\text{dtbpe})\text{MR}_2\text{Li}(\text{OR}_{12})_n$  [I; M = Rh, Ir; dtbpe =  $(\text{Me}_3\text{C})_2\text{PCH}_2\text{CH}_2\text{P}(\text{CMe}_3)_2$ ; R = Me, Ph, *o*- and *p*-tolyl, C.tplbond.CCMe<sub>3</sub>, CH<sub>2</sub>SiMe<sub>3</sub>; R<sub>12</sub> = Et<sub>2</sub>, (CH<sub>2</sub>)<sub>4</sub>] were prepared by treating RLi with  $[(\mu\text{-Cl})\text{M}(\text{dtbpe})]_2$  in Et<sub>2</sub>O and characterized by <sup>1</sup>H, <sup>31</sup>P, and <sup>13</sup>C NMR spectroscopy. I are monomeric in polar and nonpolar solvents as determined by multinuclear NMR spectroscopy. Thermally less stable

(dippe)Rh(p-MeC<sub>6</sub>H<sub>4</sub>)<sub>2</sub>Li(OEt)<sub>2</sub>1.6 [dippe = (Me<sub>2</sub>CH)<sub>2</sub>PCH<sub>2</sub>CH<sub>2</sub>P(CHMe)<sub>2</sub>]<sub>2</sub> was also prepared Crossover expts. indicate that I are inert toward exchange of their hydrocarbon ligands in solution Protonation of I (M = Ir, R = Ph, R<sub>1</sub> = Et) with H<sub>2</sub>O and I (M = Rh, R = o-tolyl, R<sub>1</sub> = Et) with Me<sub>2</sub>CHOH-d<sub>8</sub> affords [(μ-OH) Ir(dtbpe)]<sub>2</sub> and [(μ-H)Rh(dtbpe)]<sub>2</sub>, resp. The latter product apparently is formed via a series of protonation-reductive elimination sequences followed ultimately by a β-hydride abstraction from the coordinated alkoxide ligand. I also act as mild alkylating agents toward transition metal halide complexes. E.g., I (M = Rh, R = o-tolyl, R<sub>1</sub> = Et) with [(Me<sub>3</sub>P)<sub>4</sub>Rh]<sup>+</sup> Cl<sup>-</sup> in THF affords (o-MeC<sub>6</sub>H<sub>4</sub>)Rh(PMe<sub>3</sub>)<sub>3</sub> and (o-MeC<sub>6</sub>H<sub>4</sub>)Rh(PMe<sub>3</sub>)(dtbpe).

RX(5) OF 26



L4 ANSWER 42 OF 50 CASREACT COPYRIGHT 2008 ACS on STN

ACCESSION NUMBER: 105:226917 CASREACT

TITLE: Reactions of complex ligands. Part 28. Nucleophilic acylation of metal carbonyls. Synthesis and structure of a tetracarbonyl(pivaloylacetyl)ferrate and its reaction with electrophiles

AUTHOR(S): Doetz, Karl Heinz; Wenicker, Ulrike; Mueller, Gerhard; Alt, Helmut G.; Seyferth, Dietmar

CORPORATE SOURCE: Anorg. Chem. Inst., Tech. Univ. Muenchen, Garching, D-8046, Fed. Rep. Ger.

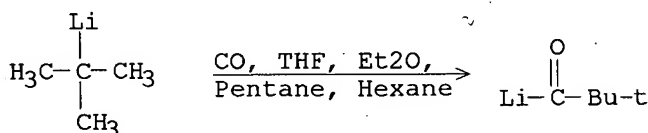
SOURCE: Organometallics (1986), 5(12), 2570-2  
CODEN: ORGND7; ISSN: 0276-7333

DOCUMENT TYPE: Journal

LANGUAGE: English

AB Nucleophilic acylation of Fe(CO)<sub>5</sub> by in situ-generated Me<sub>3</sub>CCOLi at low temperature affords an acylferrate complex, the structure of the [(Ph<sub>3</sub>P)<sub>2</sub>N]<sup>+</sup> salt of which is determined by x-ray crystallog. The acylferrate reacts with electrophiles to give thermolabile products which are characterized as tetracarbonyl acylcarbene complexes by IR spectroscopy.

RX(6) OF 21



L4 ANSWER 43 OF 50 CASREACT COPYRIGHT 2008 ACS on STN

ACCESSION NUMBER: 104:5987 CASREACT

TITLE: Synthesis and crystal structure of isomerized butadiene(dicarbonyl)(ethoxyarylcabene)iron complexes

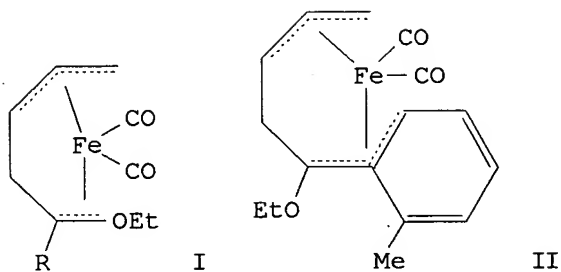
AUTHOR(S): Chen, Jiabi; Lei, Guixin; Xu, Weihua; Jin, Xianglin; Shao, Meicheng; Tang, Youqi

CORPORATE SOURCE: Shanghai Inst. Org. Chem., Acad. Sin., Shanghai, Peop. Rep. China

SOURCE: Journal of Organometallic Chemistry (1985), 286(1), 55-67

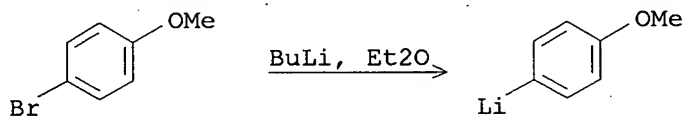
CODEN: JORCAI; ISSN: 0022-328X

DOCUMENT TYPE: Journal

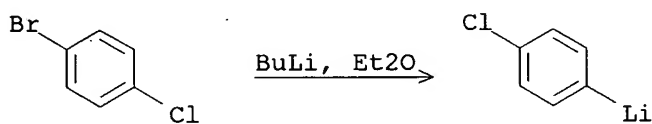


AB Reaction of  $\pi$ -butadienetricarbonyliron with RLi (R = Ph, substituted Ph, 1-naphthyl and 2-thienyl) in ether at low temperature, followed by the alkylation of the acylmetallate obtained with Et<sub>3</sub>OBF<sub>4</sub> in water at 0° gave 10 complexes C<sub>4</sub>H<sub>6</sub>(CO)<sub>2</sub>FeC(OEt). From elemental analyses, IR, <sup>1</sup>H NMR and mass spectra, as well as single crystal x-ray structure determination, these new compds. were isomers of butadienyldicarbonyl(ethoxyarylcabene)iron complexes with two types of structure I and II. A possible reaction mechanism is proposed and discussed.

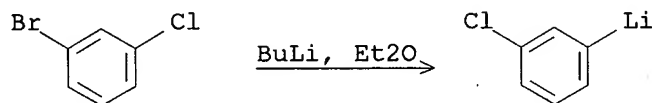
RX(12) OF 22



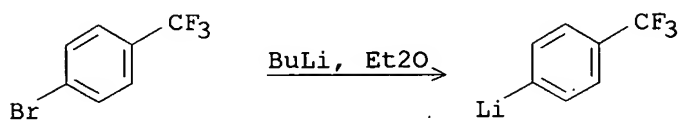
RX(13) OF 22



RX(14) OF 22



RX(15) OF 22





TITLE: Regiospecific hydride abstraction from metallacycles: conversion of metallacyclopentanes to cationic  $\pi$ -allylic complexes

AUTHOR(S): Barabotti, Paolo; Diversi, Pietro; Ingrosso, Giovanni; Lucherini, Antonio; Nuti, Franca

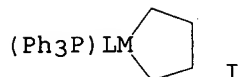
CORPORATE SOURCE: Ist. Chim. Org. Ind., Univ. Pisa, Pisa, 56100, Italy

SOURCE: Journal of the Chemical Society, Dalton Transactions: Inorganic Chemistry (1972-1999) (1984), (11), 2517-23  
CODEN: JCOTBI; ISSN: 0300-9246

DOCUMENT TYPE: Journal

LANGUAGE: English

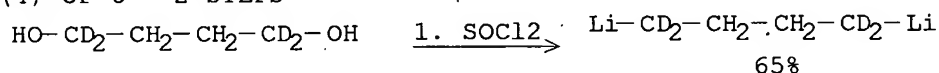
GI



AB Complexes I (M = Rh, Ir; L =  $\eta^5$ -pentamethylcyclopentadienyl) react with Ph<sub>3</sub>C<sup>+</sup> to give [MLL<sub>1</sub>(PPh<sub>3</sub>)] [BF<sub>4</sub>] (L<sub>1</sub> =  $\eta^3$ -1-methylallyl). D-labeling studies showed that Ph<sub>3</sub>C<sup>+</sup> abstrs. a  $\beta$ -H atom from I regiospecifically. The involvement of a  $\sigma$ -3-butenyl intermediate which rearranges to a  $\eta^3$ -1-methylallyl derivative is confirmed by the reactions of PdCl<sub>2</sub>L<sub>2</sub> (II; L<sub>2</sub> = Ph<sub>2</sub>PCH<sub>2</sub>CH<sub>2</sub>PPh<sub>2</sub>) or RhL(PPh<sub>3</sub>)I<sub>2</sub> (III) with CH<sub>2</sub>:CHCH<sub>2</sub>CH<sub>2</sub>MgBr. II gives a  $\sigma$ -3-butenyl complex which reacts with AgBF<sub>4</sub> to give [PdL<sub>1</sub>L<sub>2</sub>] [BF<sub>4</sub>], whereas III loses a PPh<sub>3</sub> ligand and gives RhLL<sub>1</sub>I directly. Reaction of III with MeCH:CHCH<sub>2</sub>CH<sub>2</sub>MgBr gives [RhLL<sub>3</sub>(PPh<sub>3</sub>)] [BF<sub>4</sub>] (L<sub>3</sub> =  $\eta^3$ -1,3-dimethylallyl).



RX(4) OF 6 - 2 STEPS



RX(6) OF 6 - 3 STEPS



L4 ANSWER 45 OF 50 CASREACT COPYRIGHT 2008 ACS on STN

ACCESSION NUMBER: 102:132102 CASREACT

TITLE: Vibrational spectra of alkali metal cyclopentadienides

AUTHOR(S): Garbuzova, I. A.; Garkusha, O. G.; Lokshin, B. V.; Borisov, G. K.; Morozova, T. S.

CORPORATE SOURCE: A. N. Nesmeyanov Inst. Org.-Elem. Compd., Moscow, USSR

SOURCE: Journal of Organometallic Chemistry (1985), 279(3), 327-35

CODEN: JORCAI; ISSN: 0022-328X

DOCUMENT TYPE: Journal

LANGUAGE: English

AB The vibrational spectra of crystalline alkali metal cyclopentadienides (CpM; M = Li, Na, K) and the Raman spectra of CpLi and CpNa in THF were obtained. The Raman spectra of solid CpM (M = Li, Na, K) were measured both at room and liquid N temps. The spectra were very similar to those of  $\eta^5$ -Cp complexes with a predominantly ionic metal-ligand bond. The polarity of the M-Cp bond increases upon solution. In the low-frequency range of the Raman spectra of CpLi and CpNa solns., a weak line assigned to the tilt vibration of the anion in a tight ion-pair (M+Cp<sup>-</sup>) was revealed. The dependence of the low-frequency Raman spectra of  $\eta^5$ -Cp complexes on the metal-ligand bonding is discussed.

RX(1) OF 3



L4 ANSWER 46 OF 50 CASREACT COPYRIGHT 2008 ACS on STN

ACCESSION NUMBER: 101:6668 CASREACT

TITLE: Asymmetric syntheses of (-)-cycloocten-3-ol and (-)-3-deuteriocyclooctene via nucleophilic attack on ( $\eta^3$ -cyclooctenyl)molybdenum complexes

AUTHOR(S): Faller, J. W.; Chao, Kuo Hua

CORPORATE SOURCE: Dep. Chem., Yale Univ., New Haven, CT, 06511, USA

SOURCE: Organometallics (1984), 3(6), 927-32

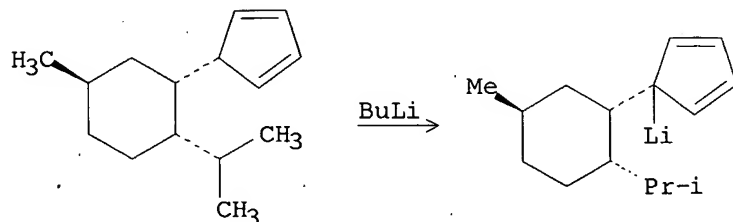
CODEN: ORGND7; ISSN: 0276-7333

DOCUMENT TYPE: Journal

LANGUAGE: English

AB Nucleophilic attack on the exo isomer of  $[R(CO)(NO)MoL]^+$  (R = neomenthylcyclopentadienyl, L =  $\eta^3$ -cyclooctenyl) provides a facile route to optically pure, allylically substituted cyclic olefins. The configuration at the metal center controls the configuration at the allylic center owing to preferential attack cis to the NO ligand in the exo isomer. (+)-(R)- $[R(CO)(NO)MoL]^+$  (I) was obtained from (S)-R(NO)MoBrL (II), which was readily separated from the diastereomeric mixture of the bromide complex. Thus, reaction of NaBD<sub>3</sub>CN and H<sub>2</sub>O with I gave (-)-(R)-cyclooctene-3-d and (-)-(R)-2-cycloocten-1-ol, resp., upon liberation of the olefin from the complex. The crystal and mol. structure of II were determined by x-ray crystallog. anal.

RX(2) OF 2



L4 ANSWER 47 OF 50 CASREACT COPYRIGHT 2008 ACS on STN

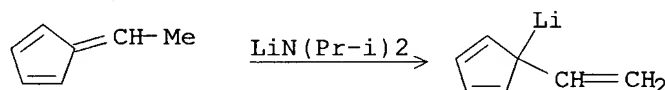
ACCESSION NUMBER: 99:176002 CASREACT

TITLE: The synthesis and proton NMR study of vinyl

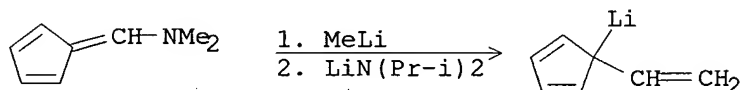
organometallic monomers: ( $\eta^5$ -  
 $C_5H_4CH:CH_2$ ) $M(CO)_2(NO)$  ( $M = Cr, Mo, W$ ) and  
 $(\eta^5-C_5H_4CH:CH_2)M(CO)_2$  ( $M = Co, Rh, Ir$ )  
 AUTHOR(S): Macomber, David W.; Spink, W. Craig; Rausch, Marvin D.  
 CORPORATE SOURCE: Dep. Chem., Univ. Massachusetts, Amherst, MA, 01003,  
 USA  
 SOURCE: Journal of Organometallic Chemistry (1983), 250(1),  
 311-18  
 CODEN: JORCAI; ISSN: 0022-328X  
 DOCUMENT TYPE: Journal  
 LANGUAGE: English

AB A reaction between 6-methylfulvene and lithium diisopropylamine in THF produces vinylcyclopentadienyllithium in yields of 85-95%. The  $^1H$  NMR spectrum of this air-sensitive organolithium reagent has been recorded in THF- $d_8$ . Reactions of vinylcyclopentadienyllithium with Group VIB metal hexacarbonyls followed by treatment with N-methyl-N-nitroso-p-toluenesulfonamide afford the new vinyl organometallic monomers  $(\eta^5-C_5H_4CH:CH_2)M(CO)_2(NO)$  ( $M = Mo, W$ ). Vinylcyclopentadienyllithium also serves as a convenient precursor to a series of ( $\eta^5$ -vinylcyclopentadienyl)dicarbonylmetal monomers of Co, Rh, and Ir. The  $^1H$  NMR spectra of these vinylcyclopentadienylmetal derivs. have been compared as a function of the metal.

RX(3) OF 4



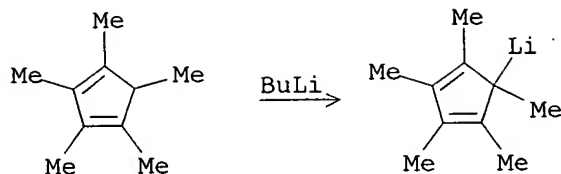
RX(4) OF 4 - 2 STEPS



L4 ANSWER 48 OF 50 CASREACT COPYRIGHT 2008 ACS on STN

ACCESSION NUMBER: 91:91738 CASREACT  
 TITLE: Syntheses and electronic structures of  
 decamethylmanganocenes  
 AUTHOR(S): Robbins, John L.; Edelstein, Norman M.; Cooper,  
 Stephen R.; Smart, James C.  
 CORPORATE SOURCE: Mater. Mol. Res. Div., Lawrence Berkeley Lab.,  
 Berkeley, CA, 94720, USA  
 SOURCE: Journal of the American Chemical Society (1979),  
 101(14), 3853-7  
 CODEN: JACSAT; ISSN: 0002-7863  
 DOCUMENT TYPE: Journal  
 LANGUAGE: English

AB The syntheses of  $[(C_5(CH_3)_5)_2Mn]PF_6$ ,  $(C_5(CH_3)_5)_2Mn$ , and  $Na[(C_5(CH_3)_5)_2Mn]$  are described. Magnetic susceptibility, IR, electrochem., NMR, and reactivity studies suggest the formulation of these complexes as low-spin 16-, 17-, and 18-electron planar metallocenes. EPR spectra of the neutral complex are consistent with the 2E<sub>2g</sub> configuration determined for other low-spin 17-electron metallocenes.



L4 ANSWER 49 OF 50 CASREACT COPYRIGHT 2008 ACS on STN

ACCESSION NUMBER: 90:151900 CASREACT

TITLE: Reactions of arylsulfonyl compounds with an excess of organolithium reagent. 13. Synthesis of o-mercaptosulfones and o-disulfones of benzene, naphthalene, and thiophene series based on o-lithiumsulfones

AUTHOR(S): Gol'dfarb, Ya. L.; Stoyanovich, F. M.; Chermanova, G. B.; Lubuzh, E. D.

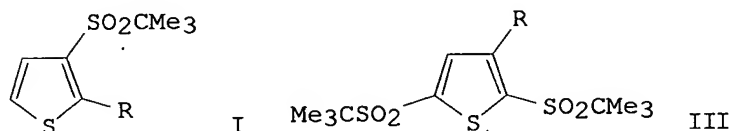
CORPORATE SOURCE: Inst. Org. Khim. im. Zelinskogo, Moscow, USSR  
SOURCE: Izvestiya Akademii Nauk SSSR, Seriya Khimicheskaya (1978), (12), 2760-7

CODEN: IASKA6; ISSN: 0002-3353

DOCUMENT TYPE: Journal

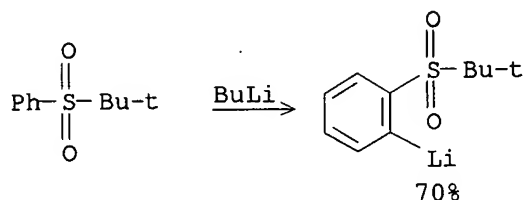
LANGUAGE: Russian

GI

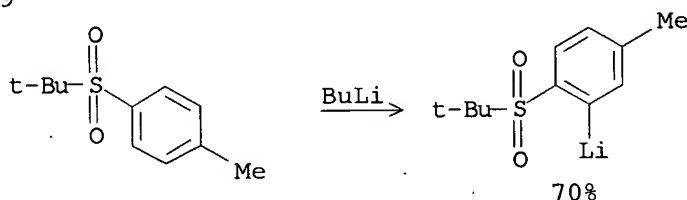


AB O-mercapto sulfones of the benzene, naphthalene and thiophenes and their transition metal salts were prepared. The IR showed the existence of the intramol. H bond in the mercaptosulfones; it also confirmed the participation of the sulfonyl groups in coordination with the metal in their metallic derivs. A series of o-bissulfones and trissulfones of the benzene and thiophene series were prepared; one of these sulfonyl groups was the principal possibility for nucleophilic substitution. Mercaptan I (R = SH) (II) was obtained in 79% yield by reaction of I (R = Li) with S. II was converted to 30-63% I (R = MeS, Me<sub>3</sub>CS) by reaction with the corresponding alkyl halides. The sulfides were oxidized to give 40.5-68% I (R = MeSO<sub>2</sub>, Me<sub>3</sub>CSO<sub>2</sub>). Treatment of thiophene III (R = H) with LiN(CHMe<sub>2</sub>)<sub>2</sub> at -40° followed by S gave 79.5% III (R = SH). III (R = SO<sub>2</sub>CMe<sub>3</sub>) was obtained by successive treatment of 3-tert-butylthio-2,5-dilithiothiophene with S, MeS and H<sub>2</sub>O<sub>2</sub> in AcOH.

RX(5) OF 49



RX(6) OF 49



L4 ANSWER 50 OF 50 CASREACT COPYRIGHT 2008 ACS on STN

ACCESSION NUMBER: 63:21450 CASREACT

TITLE: Infrared spectra of isotope-substituted molecules of ethyl-lithium

AUTHOR(S): Rodionov, A. N.; Talalaeva, T. V.; Shigorin, D. N.; Rodionova, G. N.; Kocheshkov, K. A.

CORPORATE SOURCE: L. Ya. Karpov Phys. Chem. Inst., Moscow

SOURCE: Izvestiya Akademii Nauk SSSR, Seriya Khimicheskaya (1965), (4), 604-10

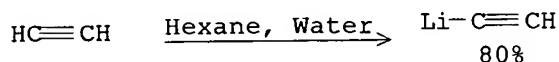
CODEN: IASKA6; ISSN: 0002-3353

DOCUMENT TYPE: Journal

LANGUAGE: Russian

AB The ir spectra of Et7Li, Et6Li, CH3CD27Li, CD3CH27Li, and C2D57Li were recorded in the 400-3500-cm.-1 region. The band at 528 cm.-1 is shifted approx. 20 cm.-1 by the ion exchange of the H atom in the  $\alpha$  position and of the Li atom, resp., whereas the exchange of the H in the  $\beta$  position has no effect. The band is attributed to the CH2-Li group in the complex, and the isotope effect is in accordance with the theory. Besides the bands in the stretching region of the aliphatic CH and CD groups, bands shifted 50-100 cm.-1 to lower frequencies are found. The bands are attributed to the  $\alpha$ -CH2 and  $\alpha$ -CD2 groups, and the shift is explained by the inductive effect of the electropos. Li atom and by the intermol. interactions. The bands in the 850-925-cm.-1 region are attributed to the stretching vibrations of the CC group. The spectra of the crystals differ from those of the vapor and of the liquid, and the effect of the isotope exchange is lower in the crystalline state than in the gaseous and liquid states. The differences are due to the changes in the structure of the complexes.

RX(1) OF 1



NOTE: Classification: Lithiation; # Conditions: n-BuLi; hexane H2O; -25 deg /Ar

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---Logging off of STN---

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Executing the logoff script...

=> LOG Y

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